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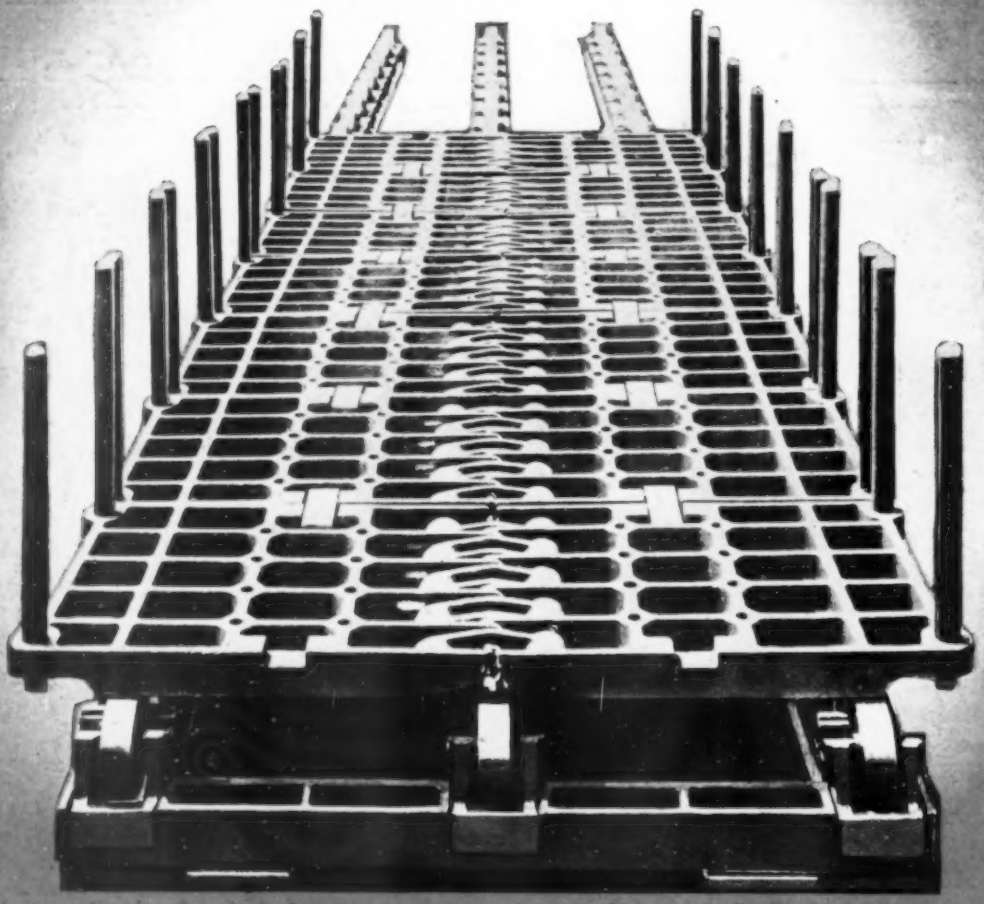

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The British Journal of Metals

(INCORPORATING THE METALLURGICAL ENGINEER)

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METALLURGIA

THE BRITISH JOURNAL OF METALS.

INCORPORATING "THE METALLURGICAL ENGINEER."

MARCH 1948 Vol. XXXVII No. 221

Greater Production—A Challenge

WE are being continually warned by political and industrial leaders and by many others that Britain is living beyond her means. This is the blunt truth and is responsible for the serious disequilibrium in her balance of payments. It is well known that Britain faces an economic crisis of great magnitude and, as emphasised in earlier issues of this journal, we believe the only solution to her problems to be greater productivity. It is true, as pointed out in the recent *Monthly Statistical Bulletin* of the British Iron and Steel Federation, that there are three lines of action open to the Government with a view to effecting a solution: curtailing the supply of consumer goods available to her own people; reduce expenditure by business firms and public bodies on capital goods of all kinds; or, seek means whereby the national products can be increased. Each of these avenues is being explored simultaneously; indeed, some of the results of a close study of these lines of action have been put into operation. The main comment of the *Bulletin* on these actions is that insufficient emphasis has been put upon the possible contribution from increased production with the corollary of an exaggerated emphasis on cuts in capital expenditure. This is borne out by the Government's economic survey, just published as a White Paper, in which many aspects of Britain's economic situation are discussed.

Britain is already committed to a considerably increased expenditure on education, health services, pensions and social services of all kinds. She cannot meet these commitments to a higher standard of living than pre-war on the basis of a production very little higher than that of a relatively poor pre-war year and with the loss of the income which we used to draw from investments abroad and for services overseas. Her urgent need is increased productivity. With an output in physical terms only about 5% more than in 1938, which was recognised as a relatively poor year, and with the loss of incomes referred to, improved performance over the whole of Britain's economy is imperative. Indeed, unless productivity in many sections of industry can be raised above the present level it will not be possible to meet these commitments without depressing the standard of living in a way which, through the reduction of incentives, with fewer consumer goods and the increase in social frictions engendered when living standards are falling, may well react adversely on production and further aggravate the economic problems. The possibilities open to British economy of raising output per head must urgently be sought throughout the whole field of production and distribution, in personal services, public utilities, the Civil Service and agriculture as well as in the field of manufacturing industry.

In a previous issue* we stated that before the wa the productivity of the United States was outstripping that of the rest of the world, and it was becoming increasingly difficult for other countries to balance their payments to that country. The enormous increase in productivity in the United States during and since the war has enabled her to build up a huge surplus of exports over imports. Her annual output of goods and services, for instance, has increased by 66% since 1938, according to the *Monthly Statistical Bulletin*. Surely, therefore, greater production is the key to Britain's economic difficulties; even when every allowance is made for the advantages of the American position, the conclusion must be reached that there is much scope for improvement.

The Government has sought to drive home the lesson that 10% more will turn the tide. Such an advance in every sector of the economy would raise the national income of some £900 million. Assuming that the right financial policy was pursued, this would be sufficient to close the over-all gap in the balance of payments without cutting capital expenditure, while still allowing a modest increase in consumption. This also assumes increased output in the right products for export or for capital investment and the solution of the dollar drain. The latter is a special problem. Although Britain is building up surpluses in the sterling areas, she is accumulating heavy deficits in dollar countries, and even the White Paper does not indicate how one can be used to effset the other.

In many branches of industry, e.g., coal, cotton textiles, building, shipbuilding and engineering, output per head is below the pre-war level, though agriculture and steel are exceptions to the rule. This fact suggests that there is ample scope for improvement.

The new policy of emphasising the importance of relating incomes to increased output should be of assistance in bringing about this improvement. In addition, between 200,000 and 300,000 additional workers may be expected to be available for productive industry this year, as compared with 1947, as a result of further releases from the Forces. Therefore, the target increase in production is well within the bounds of possibility.

The possibilities can be seen more clearly by reference to the iron and steel industry. Assuming the target output of 14 million ingot tons is achieved—the chances of the iron and steel industry's "ten per cent. more" are excellent. Production in January was at an annual rate of 14½ million tons. This is an increase of 12% over 1947, 44% over 1938. Since an additional 500,000 tons of finished steel will probably be needed by the metal-using industries to meet their export targets, a similar additional quantity should be available for purely domestic purposes. The larger output of steel should

* October, 1947.

sustain a more proportionate increase in the output of engineering and other industries whose "work in progress" piled up last year. But, while a larger output is possible, it depends on an adequate supply of scrap.

Prospects of similar increases in other branches of industry are favourable, especially in view of the improvement in coal supplies. Thus, production has lately been making headway in the cotton and wool textile industries, while in agriculture substantially larger crops should be secured if weather conditions are favourable. In short, ample scope exists for an improvement in productivity in many branches of the British economy. To devise means of exploiting all these possibilities to the full should be the primary aim of the Government, of managements and of organised labour. There should, however, be a keen recognition by all of the hard fundamentals of British economy, greater productivity would then become a challenge.

The Theories of Creep in Metals

A Summer School in Theoretical Physics, with particular application to creep in metals, will be held in the H. H. Wills Physical Laboratory of the University of Bristol beginning 10 a.m., June 2nd, and ending June 5th, 1948.

The course is similar in conception to that already held on the electrical properties of solids in June, 1946, and on mechanical properties of metals in July, 1947. It is intended mainly for members of the staffs of Government and industrial laboratories who wish to familiarise themselves with the theoretical side of subjects with which they have been concerned on the experimental side. Lectures will be given by Prof. N. F. Mott and other members of the staff of H. H. Wills Physical Laboratory. In addition to the regular lectures, there will be opportunities for research workers attending the course to discuss their own work and its theoretical implications.

The subjects to be treated will include: Anelastic effects due to slip at grain boundaries; grain growth, recrystallisation and the theory of grain-boundary slip; geometry of dislocation and the theory of slip bands; theory of transient creep; slip in solid solutions; diffusion of carbon in iron and elastic after effect; effects of carbon on the mechanical properties of iron; mechanisms of internal damping; crystal growth and the origin of dislocations.

The number attending the course will be limited to about 50. The fee for the course is 5 guineas. Further particulars and forms of application, to be returned by April 15th, can be obtained either from the Director of the Department of Adult Education, The University of Bristol, or from the Secretary of the Institute of Physics, 47, Belgrave Square, London, S.W. 1.

Associazone Italiania di Metallurgia

Owing to unexpected difficulties the Second National Meeting of the above Association has been postponed. Arrangements have now been made for the meeting to be held in Milan on April 23rd to 25th inclusive, which coincide with the XXVI Exhibition. The subjects to be discussed on the first day of the meeting include the corrosion and the ageing of metals; the remaining two days are given over to open discussion on various subjects. The time limit for summaries of communications to the meeting is February 29th; for the whole text March 31st. Inquiries should be made to the Associazione Italiania di Metallurgia, S. Paolo, 10, Milano, Italy.

The Protection of Steel against Atmospheric Corrosion and Marine Fouling

A joint meeting of the Iron and Steel Institute and the British Iron and Steel Research Association to discuss several papers dealing with the protection of steel against atmospheric corrosion and marine fouling will be held at the Institute's offices on June 17th, 1948. Mr. T. M. Herbert, deputy chairman of the Corrosion Committee and chairman of the Protective Coatings Sub-Committee, will preside at the morning session, and Professor J. E. Harris, Chairman of the Marine Corrosion Sub-Committee, at the afternoon session.

At the morning session, commencing at 10-30 a.m., the "First Report of the Methods of Testing (Corrosion) Sub-Committee," is to be presented by the Chairman, Dr. J. C. Hudson, after which a joint discussion will be held on: "Service Trials of Painting Schemes Applied to a Steelworks Gantry," by Dr. J. C. Hudson and "The Protection of Iron and Steel by Various Non-Metallic Coatings," by Dr. J. C. Hudson and Dr. T. A. Banfield.

At the afternoon session, commencing at 2-15 p.m., a joint discussion will be held on "Studies on Anti-Fouling Compositions, Parts I to III," by Dr. H. Barnes; and "Service Tests of Experimental Anti-Fouling Compositions," by Dr. H. Barnes, M. W. H. Bishop and K. A. Pyefinch. The session will conclude with a joint discussion on "Cementiferous Paints," by Dr. J. E. O. Mayne and Dr. R. S. Thornhill; and "Marine Exposures of Cementiferous Painting Schemes," by K. A. Pyefinch.

Non-members of the Institute will be welcome to come to the meeting and to join in the discussion. Separate reprints of the papers are obtainable for 2/6 each and Buffet Lunch tickets for 6/- each, or a complete set of the seven papers and a buffet lunch ticket may be had for £1. Applications accompanied by a remittance should be sent to the Secretary of the Iron and Steel Institute (4, Grosvenor Gardens, London, S.W.1), to arrive not later than Monday, June 14th (envelopes to be marked "Corrosion Meeting").

Metal Flow Research

The British Iron and Steel Research Association has now obtained a licence for erecting limited accommodation of temporary construction on a site at Meadow Street in Sheffield. This temporary construction will provide some further accommodation for the expanding programme of research on metal working problems.

At the present time, B.I.S.R.A's investigations at Sheffield are widely centred between Sheffield University, Applied Science Department (where there is an Experimental Rolling Mill), the works of Arthur Lee and Sons Ltd. (where there is a Prototype Backpull Wire Drawing Machine) and elsewhere.

The intention is ultimately to create a permanent building in which rolling, drawing and other similar researches will be centralised. It is hoped that these new Metal Flow Laboratories will be one of the world's best equipped laboratories for their purpose.

This will be the sixth of B.I.S.R.A's "outstations," the others being: The Corrosion Laborary at Birmingham: The Marine Biological Laboratory at Scotland: The Experimental Blast and Open-Hearth Furnace at Stoke-on-Trent; The South Wales Laboratories at Swansea, which is described elsewhere in this issue; and The Physics Laboratories at Battersea, London.

METALS and MANKIND: from ANGLESEY to HIROSHIMA

By Hugh O'Neil

Professor of Metallurgy, University College, Swansea

In his recent Inaugural Lecture, given at the University College of Swansea, Dr. O'Neill referred to hisr pedecessor, Dr. C. A. Edwards, F.R.S., appointed in 1921, *as the first Professor of Metallurgy of the College, and whose retirement in* 1947 *brought to a close a period of most fruitful activity as University teacher and researcher. The long list of nearly fifty published papers from this Metallurgical Department bears witness to some of the work which Dr. Edwards inspired and encouraged whilst in South Wales ; he felt that all would join him in wishing the Emeritus-Professor many happy years free from administrative cares. His Lecture, which is presented here, covered a wide field from isolated periods connected with the development of metalworking in Britain, to the new era opened by the atom bomb, with its consequences for good or evil. The key to the latter is with mankind and must be used to effect a peaceful future for the atomic energy of metals.*

Introduction and Summary

THE importance of metallurgy shows no signs of diminishing, for we are still in the Age of Metals. The ambition of the late Henry Ford actually to " grow " a motor-car is a reminder that organic compounds known as " plastics " may replace metals to some extent, yet the approach of an era where power will be obtained from metallic atoms considerably increases the responsibilities of metallurgists. Furthermore, the necessity of reducing production costs in our factories means a demand for more efficient processes. The training of metallurgical students must therefore be undertaken with these two prospects in mind.

Historical considerations have prompted the title of this lecture, for the history of a technical subject forms a link with its human aspect. It may be helpful if I summarise the ground which will be covered. I propose to give some particulars of a few isolated periods connected with the development of metalworking in Britain. Emphasis will be placed on the metallic property of ductility, for craftsmanship has relied upon it from very early times. The results which have been obtained leave us marvelling at the inherent capabilities of mankind, but unfortunately swords are always in demand as much as ploughshares, and destructiveness cancels much of the progress which is made. Praiseworthy researches in universities about the structure of the atom and the reason *why* metals are ductile have finally left us with that ambiguous gift, the atomic bomb. The conclusion may be drawn that the present crisis of our Metal Age is not brought about by any shortcomings of physical science. It is due to a poor technique on the part of mankind in adopting rules which are necessary for the Good Life. Since the universities have given us the atomic bomb, they should equally exert themselves as civilising influences to counteract its possibilities for evil.

I have said that metals entered the service of man because of their ductility. Gold for instance, may be beaten out into leaves only 1/300,000 of an inch in thickness. The cold rolling of steel is a great industry in South Wales which depends upon this property. There is something paradoxical about the metallic state for whilst it chiefly implies plasticity and toughness, yet it also suggests durability and hardness. A given piece of metal may sometimes possess either type of property depending upon its previous treatment. This is why metal replaced flint and bone, but since it was pleasing in appearance and scarce at first, the initial use was for precious ornaments. Almost at once came the production of cutters and hammers and axes in bronze. We then encounter the mixed nature of man who will employ raw materials indiscriminately for tools, weapons, ornaments and the accessories of religious practice. Throughout the ages the arts of war and peace have competed with each other, and treasures and monuments painstakingly acquired are lost periodically as a result of conflicts. A primitive metal smith will one day hammer-out a lump of ductile iron, bend-over the sheet and rivet it to make a handbell. Next day he will fashion a bronze casting to produce a sword. The medieval foundrymen who cast the large church bells eventually modified the design and produced the first great mortars for the armies. The artillery contest of the irresistible projectile and the impregnable defence is still with us, and new hard cutting-alloys which will turn glass in lathes as though it were cheese now find employment as anti-tank bullets. Natural philosophers have busily speculated for centuries on the ultimate nature of metals, and unitary theories of matter, backed by the needs of princes, prompted the quest to transform base materials into the " perfect " element—gold. Men of science have at last succeeded in effecting various transmutations, from which it has been but a philosophical stone's throw to atom-splitting or nuclear fission. The atom bomb burst open a new era with consequences for good or evil which remain to be seen, and now the race is on for metallic uranium which is probably the key strategic material in the world to-day. This is the picture for our further study.

Ancient Welding and Early Tinplate

You will remember that the Bard in Gray's famous poem saw from a mountain in North Wales the pageant of the past and the shape of things to come. As a starting place I would invite you to Holyhead Mountain, for Anglesey has special points of interest. During a holiday there I came across at Ty Mawr, on the western slopes of the hill above South Stack, the remains of some prehistoric circular huts. One of these proves to be the dwelling place and workshop of a metallurgist who flourished during the second millenium before

Christ. This was the time when Ireland was beginning to produce flat copper axes and beautiful ornaments of pure gold, and on very clear days our smith would be able to see the Wicklow Hills which were the source of all this remarkable wealth. Anglesey lay on a sea route which made it susceptible to Irish and Scottish influences, and the dawn of the Age of Metal in Wales depended upon geographical factors.

The traffic from Ireland included the lunulae or "little moons" of thin gold about 8 inches across which adorned the necks of very important persons. The distribution of finds of gold ornaments during the Bronze Age shows that some were discovered in Anglesey and one in Glamorgan. An archeologist's reconstruction* of Ty Mawr workshop shows, built into the wall, a small bellows furnace having a well at the bottom about 1 ft. diameter and two inches deep. Charcoal and pounded ore produced a cake of bronze in the well which was subsequently melted in crucibles. Close beside the furnace was the casting pit to accommodate a mould for either axes, spear-heads or swords. A fire towards the centre served for the smaller operations and for domestic purposes, whilst on a work-bench located near the door, the cast metal was hammered into sheet with stone tools. The smith probably travelled from one chieftain to another and carried his tools, together with a supply of metal upon which to work.

Anglesey has recently yielded some more metal objects of this early period. During 1943, a new aerodrome was being made near Valley, and the work required the removal of peat from a bog at Llyn Cerrig Bach. An inspector noticed that the local farmer was towing his agricultural machinery with a curious iron chain. It transpired that this had been recovered from the bog, and expert examination indicated prehistoric metal. Eventually, the peat yielded about ninety objects (probably not of local origin) of the 1st or 2nd century B.C., including swords, spears, parts of vehicles, horse furniture, welded slave gang chains, iron currency bars (a kind of coinage) and a trumpet from Ireland.†

A glance at Table I shows that the slave chain is much higher in phosphorus than Roman irons of a somewhat later period.

TABLE I.—CHEMICAL ANALYSES OF ANCIENT IRON SPECIMENS

	British Slave Gang Chain ex Anglesey	Roman Bloom† ex Corstopitum nr. Hexham	Roman Chisel ex Chesterholm*
Carbon	0·07%	0·06%	0·1-3%
Silicon	tr.	tr.	0·038
Manganese	tr.	tr.	tr.
Sulphur	tr.	0·008	0·011
Phosphorus	1·05	0·063	0·016
Remarks	Probably made in East Anglia. Welded.	Weighs 3 cwt. Welded in sections.	8 in. long. Steely zones. Not carburised. Martensitic patch at part of chisel edge Hp = 579

† *J. Iron and Steel Inst.*, 1912, **85**, 118.
* *Proc. Univ. Durham Phil. Society*, IX, p. 141.

A cast bronze chariot axle cap bore the ancient emblem of the swastika—but we may surely consider it as denazified after a submersion of twenty centuries. There were some iron tyres—probably for chariot wheels. One of them is 2 ft. 6 in. in diameter and 1·5 in. wide and consists of a strip of metal which had been joined by welding. An ornate horse bridle bit of wrought iron had been coated all over with bronze by the casting-on process. The work had probably been done in Gloucestershire and should interest those of us who live in the tinplate area. A site at Bredon Hill has yielded similar prehistoric bridle bits of iron coated with tin—an anticipation by eighteen centuries of the subsequent introduction of tinned iron into the Severn region. Some iron currency bars were also found in Anglesey. Nearly 2,000 such bars are known and it is interesting that these new finds prove to be of one of the previously recognised standard weights of 300 grams.

About 500 B.C. the Irish gold trade was waning but objects of copper alloy continued to be made in Wales. It was around this time that bronze and iron working took place at Merthyr Mawr near Bridgend, and bronze razors of this period have been found in the region. After the Roman invasion, the occupying power in both South Wales and Anglesey carried out considerable metallurgical operations, and its great exploitation of the copper ore in Parys Mountain near Amlwch reminds us of this once-famous source of Welsh copper. The local copper smelters at Neath imported the Anglesey ore from 1768 onwards, but the traffic which reached its zenith in 1787 (4,000 tons output of copper) has now ceased, and the derelict plant at Amlwch is a sad reminder of a once-flourishing industry. The invaders from the north who descended upon Anglesey after the departure of the Romans, left behind some metallurgical objects of the 6–9th century A.D. comprising silver brooches and armlets.

Cornwall and Tin

Finds of the Bronze Age are very extensive and are fairly evenly distributed all over Britain. * Appreciable quantities of tin ore would be needed for making the alloy, and this must have come from Cornwall. Whilst admitting the existence of prehistoric workings in that county, examination shows that there is practically no justification for the belief that the Phoenicians from the Eastern Mediterranean traded with Cornwall for the metal. Their supplies came from "The Cassiterides"—the tin islands, but it is the identification of these which is the problem. A suggestion that one of them might be St. Michael's Mount applies almost as well to Mont St. Michel in Brittany. The documentary evidence for the Cornish tin mines only dates from 1156, so we are left to archeological evidence and speculation. The protagonists of the Phoenician story eliminate various possible sources for that traffic of knuckle-bone shaped ingots which went on through Marseilles, and they are finally left with Cornwall. It seems however, that Brittany and Spain would do just as well. Julius Cæsar mentions that the Britons smelted tin, but states that it was done in the midland districts, and apart from one single known Roman ingot of the metal found at Carnaton there is no evidence that the Romans exploited the Cornish deposits to any great extent.

The later discovery of tin in Cornwall is said to be due to St. Perran† who was born in Cork or Ossory about 350 and settled near Padstow. A black stone used in his hearth was found to have "melted" and produced a white metal. From this simple reduction process a method of quantity production was devised, and Perran became the patron saint of Cornish tin miners with a feast day on March 5th.

Another metallurgical patron is St. Dunstan, who commenced his work at Glastonbury. After appointment as Archbishop of Canterbury late in the 10th

* Maryon, H., *Proc. Roy. Irish Acad.*, 1938, 44, 181.
† See "*Interim Report*," Sir Cyril Fox, Nat. Museum of Wales, 1945, Cardiff

* "Historical Geography of England," H. C. H. Darby
† See *Nature*, 21/1/28 and 3/3/28.

Shrine of the Bell of St. Patrick's Will in gold. (A.D. 1091) Note the cross and swastika pattern, and the interlacing metal work at the side. Reproduction one-third actual size.

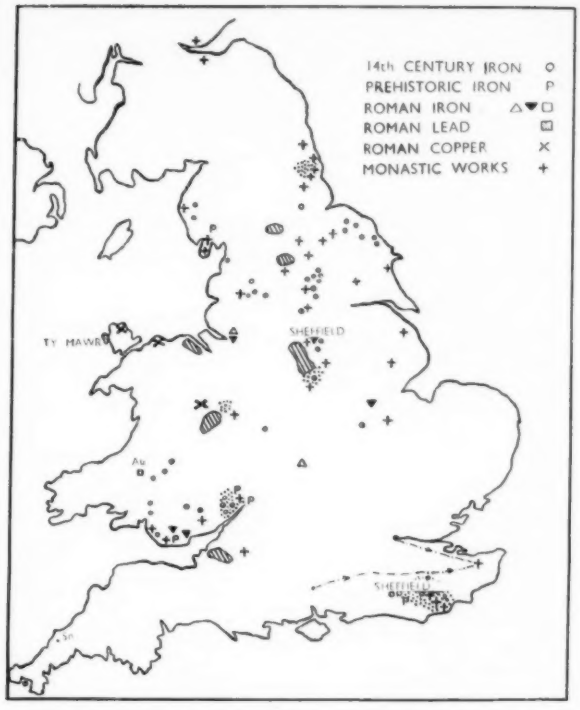

Fig. 1.—Monasteries associated with metallurgical work in Britain during the Middle Ages, together with sites of pre-historic and Roman workings. (The Pilgrims way from Winchester and London to Canterbury is shown as a dotted line)

Century, he lived at Mayfield amongst the Sussex ironworkers. In the palace are still preserved his anvil and hammer and the tongs with which, according to the Saxon Chronicle, he fought the devil:—

"St. Dunstan so the story goes,
Once pulled the devil by the nose
With red hot tongs which made him roar,
So he was heard three miles or more."

The Goldsmiths Company of London still relies upon the saint to keep the devil in his proper place. The City Companies were always imbued with a religious spirit, and we may note that "God, the only Founder," is the motto of the Founders Company.

Monastic Mining and Metallurgy

In his recent inaugural lecture, the Professor of Education paid a tribute to the cultural work of the monasteries in Wales during the Middle Ages. It is not generally appreciated that these religious foundations also played a considerable part in the development of mining and metallurgy. When the barbarian hordes overthrew the Roman Empire and swept across Europe, it so happened that Ireland escaped and remained an undamaged power-house of culture. From the time of its conversion to Christianity until about the 11th century, the western isle maintained monasteries which were not only seats of learning but centres of metal-work. The Celtic missionaries who then brought Christianity eastward to Wales, Scotland, Northumbria, France, Switzerland and North Italy also carried with them their Irish metalcraft. "Hibernian art at its best was, even in matters profane, essentially a monastic phenomenon," states Adolf Mahr.* An example of this splendid work is the 11th century container for St. Patrick's bell, shown in Fig. 1. Early monastic handbells from Wales of a similar shape and probable origin, are preserved in the National Museum at Cardiff.

We should therefore, expect the subsequent development of the Benedictine houses in Britain to lead to renewed metallurgical activity, especially since the author of the Benedictine rule expressly stated that his followers were to look upon themselves "as true monks, when they have to live by the labour of their hands." The Cistercians were associated with much of the mining work in the Pennines especially in the 14th century, for this congregation had been engaged in making iron and steel since the 11th century, at the Grande Chartreuse in France.† The cross and globe trade mark seen on bottles of Benedictine liqueur are also to be found on the medieval iron and steel products which issued from this great monastery.

The earliest reference to iron mining in Wales, for which I am indebted to Mr. Wyndham Wilkin, is an indenture dated 1253, granting to the monks of Margam Abbey "all manner of iron and lead mineral" on lands at Cornelly, near Pyle. There is a similar grant to the monks at about the same date involving an annual rent of a coulter and a ploughshare as long as they use the mineral. It seems probable that the monastery would make the ploughshare and pay it to the landlord. Another grant gave the monks the right to obtain

* See Mahr, A., "Christian Art in Ancient Ireland," Dublin; also Hudson, D. R., *Metallurgia*, April and May, 1945; and "Celtic Art in Pagan and Christian Times," J. Romilly Allen, London, 1904.

† Brangwyn, J., "Reasons for France," 1939.

mineral coal. Charcoal, however, was used for smelting and the "bloomeries" were sometimes portable and were moved about amongst the "bell pits" from which the ore was obtained. The refined iron was at first forged by hand after heating in a hearth fitted with hand bellows as may be seen in a 12th century drawing of blacksmiths.

A list which I have collected of nearly fifty religious houses engaged in mining and metallurgical work in Britain during the Middle Ages is given in Appendix A. The location of each monastery associated with such operations is shown in Fig. 2 and the sites of pre-historic and Roman workings are also marked. It is probable that the one grew successively out of the other. The monasteries and churches also needed bells to ring the hours for Office, and monks were directly associated with this type of metal founding. The bells were sometimes cast in the churchyards or even in the church or sacristy itself.

I think it is fair to say that, although these Middle Ages were lacking in the amenities which we know to-day, they were permeated by a philosophy which gave unity and meaning to life. The churchmen founded the universities and maintained an integrated connection between the different Faculties which it is generally regretted is being lost in Britain to-day. They took a long view of things—even in metallurgical affairs. There is a hint* that in order to preserve the woods which yielded charcoal for smelting, a conservative policy of woodcutting was adopted. After the Dissolution, the new landlords continued to get rich quickly by burning up the forests and thus landing the trade in difficulties. Similarly, although barbarous conflicts were plentiful in medieval times and Churchmen went to war, yet they succeeded in mitigating some of its horrors by encouraging chivalry and instituting measures like the Peace of God (990) and the Truce of God (1027), which imposed limitations on the destruction. It has been left for our times to sink back to total war.

Sheffield in the Middle Ages

War and peace are associated with cutting tools and my native Sheffield has a long-standing reputation for cutlery. Ordinary tools in medieval Britain were made from various local irons, but steel for the finer tools and weapons was imported.† A high standard of cutlery manufacture was attained in Northumbria in the 8th century.‡ Sheffield believes that its fame goes back to an entry of 1041 in the Priory Wardrobe of the Tower of London, which refers to "Cultellum de Sheffield." Then again, there is the 14th century Miller in the Reeve's tale of Chaucer who is a noteworthy person amongst the Canterbury pilgrims because "a shefield thwytel bare he in his hose." Metallurgical references to Sheffield at those times are somewhat ambiguous, for whilst the map (Fig. 2) shows Sheffield in Yorkshire (Hallamshire district), there is also a second one in Sussex, exactly on the meridian of Greenwich and seven miles north of Lewes. To-day, it is a small spot reached from Sheffield Park Station on the Southern Division of British Railways. Digression may be of interest by comparing the claims of these two places as being famed for cutlery in either the 11th or the 14th centuries.

There is a shortage of records about Hallamshire, but there are plenty of documents about the Weald. We know that horse-shoes, nails and bars of iron went up to London from Sussex at an early date. We saw that the finds in Anglesey included prehistoric iron tires for wheels, and a trade in these articles also flourished in the south. The Guild of Feroners (Ironmongers) of London, lodged a complaint about 1300 against the smiths of the Weald for making and selling iron tires too short for use. The result was action which forestalls our British Standards Institution; for it was ordered that standard length rods were to be set up in the markets.

As regards the necessary metallurgical materials, there was plenty of wood in both districts, and certainly plenty of furnace lining refractory in the north. Phosphoric iron ore of poor quality existed at the two sites, and prehistoric iron slags have been found in both places. There was only one Roman bloomery at Templeborough near Sheffield, but twelve have been traced amongst the extensive ancient workings in the Weald.

It is sometimes suggested that Sheffield on the Don in Yorkshire and nearby Rotherham on the Rother, were natural centres for cutlery because of abundant streams for working waterwheels to blow furnaces, forge metal and turn grindstones. But Sheffield on the Ouse in Sussex, and nearby sites on another adjacent river Rother, were equally well provided with waterpower. In any case, the forge at Byrkeknott (Durham) as late as 1408 is the first definite example of a water-driven hammer—though Domesday Book records what may be four water mills for iron at Lecheswrde in Somerset, and there is a French reference to "moulin a fer" in 1249.* Saltzmann† states that bellows were generally worked by hand prior to the 15th century, so that the waterpower argument is not convincing. A power-hammer is known to have existed in 1496 at Newbridge (or perhaps at Steel) in the Weald, whereas the smithy wheel at Norton for example, near Sheffield, dates from 1560.‡ There must have been earlier ones, and after the adoption of waterpower in the late 15th century, there was a phenomenal rise of the Hallamshire cutlery trades. This, of course, suggests that there had been some previous experience of the work.

There was a traffic in high quality Swedish bar iron to South Yorkshire in the Middle Ages, and it has been suggested that this material was turned into steel locally by carburising in small cementation furnaces. Unfortunately, the earliest definite mention of a Steel works is 1523 and in Sussex, with several "steel" furnaces reported at Robertsbridge in Sussex in 1609. Ellyot and Meysey obtained a patent for converting iron into steel by a cementation process in 1614 and established works near London, where "at this day probably the best and finest knives in the world are made," as a contemporary put it. This may be painful reading for people from the banks of the Don, but one must note that Overseers of Cutlery were appointed for London in 1344 and a charter was conferred on its Cutlers Company in 1415. It was not until 1624, and after overcoming objections from the City, that the Cutlers Company of Sheffield and Hallamshire was incorporated. Its earliest ordinances date from 1565 and the movement was in existence before that date.

From the beginning of the 13th century, the Weald rapidly began to supplant the Forest of Dean as the

* Schubert, H. R., *J. Iron and Steel Inst.*, April, 1947.
† Schubert, H. R., *J. Iron and Steel Inst.*, Dec., 1947.
‡ Schubert, H. R., *J. Iron and Steel Inst.*, Sept., 1947

* Straker, E., "Wealden Iron," 1931.
† *Engineering*, 1948, **165**, 165.
‡ "English Industries in the Middle Ages."

great iron-producing district, and Sussex was our Black Country in the Middle Ages.* Its Sheffield mills had eight men working in them in 1549 and 23 in 1594. High quality products could have been produced, for a considerable amount of good Spanish ore was imported into the district from an early date. Although the Poll Tax records of Richard II for 1379 note one cutler resident in "Sheffield," and Leland (16th cent.) found long-established cutlers in Rotherham and Sheffield, yet there is a strong suspicion that the knife carried by Chaucer's miller had come from the Sussex district only a few miles distant from the Pilgrim's Way.

Iron and Steel in South Wales

There is a link between the Weald and Glamorgan following upon the Dissolution of the Monasteries, for the Abbey of Robertsbridge in Sussex was given in 1541 to Sir William Sidney, father of the poet. He started ironmaking there and imported some German workmen. The venture was not very successful and in 1565 they were brought away to premises at Pentyrch, near Cardiff. In Elizabeth's time, a gun founding and iron casting industry developed in Taff Vale and Caerphilly, the "charcoal iron" industry appeared in Monmouthshire in 1558–1603, and tinplate was introduced in 1665–1720. The old mine workings af the Weald and South Wales were very similar in type, apart from practices in the latter district known as "hushing" and "scouring."† The 17th century saw a great set-back in iron production all over Britain owing to the denudation of the forests, but local activity was renewed about 1770 when pit coal replaced charcoal. Mention may be made of the old Welsh Bloomery process in Glamorgan and also of a method‡ of converting iron to steel by passing it nine times through a fire composed of charcoal, horns, hoofs and bones. This was associated with the saying :—

> "Tri chaled Byd, y maen Cellt, Dur naw Gwynias, a chalon. Mab y Crinwas";

which has been translated :—

> The three hardest things in the World—a flint stone; the steel of nine fires; and the heart of a miser.

The components of this fire are those of a good carburising mixture such as is used in case-hardening, and it is easy to appreciate that cemented steel could be produced in this way. Finally, there came Dr. Siemen's melting furnace at Landore, producing soft open hearth steel to replace iron (1875), and to-day, we have the developments of the Steel Company of Wales.

Physical Metallurgy

Our metal smith in Anglesey may well have pondered over the reason why his bronze behaved differently from his stone hammer, and we are still wondering about it do-day. The science of metallography started in 1864 and eventually showed that a piece of metal consists of a conglomerate of crystals. The spangles on the surface of galvanised steel sheet are crystals of zinc and the crystalline nature of the basis steel itself may be demonstrated with the microscope. By using beams of X-ray light it is also possible to obtain rainbow-like photographs which tell how the atoms are packed together in the metallic crystals. The science has now reached a stage where atomic physics is a most important

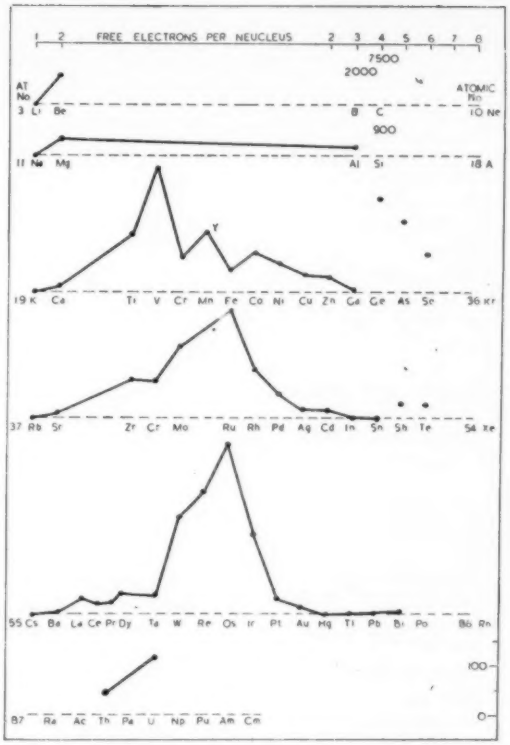

Fig. 3 Brinell hardness Vs atomic number.

consideration, and in enquiring as to the cause of ductility and electrical conductivity we have learnt a great deal about the atoms themselves. We know that the central positive nucleus of each atom is surrounded by systems of negative electrons. The nature of the electron system determines the physical properties of the atoms and of the metallic crystals built up from them. We know of nearly one hundred elements more than half of which are metals, and they can be set out in families or groups on the basis of their atomic numbers, i.e., on the number of electrons associated with each particular atomic nucleus. When we plot the measured hardness of the metals on this basis, a periodic behaviour is observed; a rise and fall of hardness in each group (Fig. 3). Notice that magnesium has 12 electrons per atom with an indentation hardness of 30 kg./sq. mm. and aluminium with 13 electrons per atom has a hardness of 16. At the bottom of the list comes uranium, which has 92 electrons, is eleven times heavier than magnesium and has a hardness of 120. Alloys of magnesium and of aluminium can be manufactured which have a strength and hardness roughly equal to that of soft steel, and we can use them in building an aircraft capable of carrying bombs. Uranium can be manipulated so that it will rapidly release enormous energy and thus act as an explosive. The aeroplane and the uranium came together over Hiroshima on August 6th, 1945, the £500 million bomb was exploded, 80,000 people soon died as a result, and Japan was brought to its knees.

The explosion came from a rapid chain reaction involving nucleur fission. By bombarding atoms of uranium 235 with atomic projectiles called neutrons, the uranium nucleii split apart with the release of great

* Delany, M. C., "The Historical Geography of the Wealden Iron Industry," 1921.
† Topley, "Geology of the Weald."
‡ Tradition recorded by Edward Williams (1812) in the Llanover MS. in the National Library of Wales. In the early 17th century the art of making steel was kept a secret.

energy, injurious gamma radiation and some more bombarding particles. The latter pass on and repeat the fission process, which rapidly builds up and cascades into an explosion. It is something like dropping a lighted firework into a box of fireworks.

If the process is controlled and slowed down, then the energy can be used for heat and power purposes, and the radio-active products are also valuable. Gigantic factories are needed for preparing the materials, and a water-cooled uranium graphite pile, with its shielding, weighs many thousands of tons. The gigantic Oak Ridge Plant, Tennessee, which incorporates the Clinton National Laboratory, makes uranium 235 from the ordinary metal, and has a staff of 43,000 people. It has been stated that radio-active materials produced here might increase agricultural productivity enough to outweigh the cost of development up to now.

It is, surely, a hopeful sign that the United States Government on January 1st, 1947, lifted the atomic energy organisation right out of the military atmosphere by handing over the Army's "Manhattan District Organisation" to a purely civilian Board. The Atomic Energy Commission of the United Nations has also shown its concern with the necessity for research and education in connection with the new processes. Since January 1st, 1948, the Oak Ridge Plant has therefore been taken over by the University of Chicago for peace-time service. The Los Alamos Plant in New Mexico is now being operated by the University of California. Nine associated eastern universities and twenty-nine participating mid-west universities are controlling the Brookhaven and the Argonne National Laboratories, respectively. The U.S. colleges are thus being given a privileged place in developing the atomic processes.

We must remember that there were great outcries at the introduction of the long bow and of gunpowder, but the atomic weapon is much more potent as a means of wrecking our civilisation. The existence of various Associations of Atomic Scientists shows that technical men are not indifferent to the tremendous dangers which now exist. It is for mankind to set its course, perhaps at least by an attempt to abandon "total war" and agree once more upon limits to the extension of military operations. One noteworthy point about the last conflict was that poison gas was not employed. The Universities of Yale and Chicago have already taken a lead in preparing plans for international control and our colleges could play a part, not by hasty resolutions about refusing to fight for King and Country, but as realistic individual centres of culture and leadership.

At the beginning of this lecture, we noticed a Welsh poet who, from his lonely mountain height, denounced the conqueror and the destructive work of warriors. We must also note that the closing lines of the Bard breathed a note of optimism which should apply to-day. The killing flash released at Hiroshima by the Allied leaders almost obscured the sun by its brightness, but we must stretch the final words of the poet to suggest a peaceful future for the atomic energy of metals :—

"... think'st thou, yon sanguine cloud
Raised by thy breath, has quenched the Orb
of day ?
To-morrow he repairs the golden flood,
And warms the nations with redoubled ray."

APPENDIX A

References to Metallurgical and Mining Activity in Britain Connected with Monastic and Religious Establishments

Establishment	Order	County	Notes
Bath, Bishop of ..		Mine in Somerset	Richard I granted lead mine.
Battle Abbey	Benedictine ..	Sussex	Owned iron working sites. In 11th century had cast bells probably made in grounds.
Beauchief Abbey ..	Premonstrat ..	Derbyshire ..	12th century. Coal and iron workings.
Beauvale Priory ..	Carthusian ..	Notts.	Leased coal mine at Newthorpe.
Beverley		Yorks.	One of the monks was a fine goldsmith.
Bolton Priory	Austin Canons	Yorks.	1294, obtained sea coals for their *forge* near Colne.
Bridlington Priory ..	Austin Canons	Yorks.	Worked iron in 12th century at Blubberhouses in Beamsley.
Byland Abbey ..	Cistercian ..	Yorks.	1180, iron mine at Denby.
Canterbury, Abbey of St. Peter of			689, received land "in which there is iron ore."
Conishead Priory ..	Austin Canons	Lancs. .. .. (Lake District)	Iron workings.
Crowland Abbey ..	Benedictine ..	Lincs.	
Darley Abbey	Austin Canons	Derbyshire ..	Charters show iron worked in 1270, Pentrich Wood, Ripley.
Dunfermline Abbey ..			1291, charter to work coal at Pittencrieff.
Durham Priory ..	Benedictine ..	Durham	12th century lead mine.
Durham, Hugh Pudsey, Bishop of ..			Later half 12th century. Iron mine at Rokehope for making ploughs.
Durham, Bishop Hatfield of			1354 coal mine. 1356, Bishop had five mines on lease to Rector of Whickham. 1492, Whickham mine had a water lift.
Durham, Bishop Langley of			1408, John Dalton, first known Durham iron-master, worked a forge for the Bishop.
Cardinal Wolsey, Bishop of			1528, built furnace to smelt lead with coal. Fell from power before test could be made.
Evesham Abbey ..	Benedictine ..	Worcester ..	Walter of Odyngton in time of Henry III gave earliest instructions for *Bell* founding, and bell size for various notes.
Finchale Priory ..	Benedictine ..	Durham	Horse pump at Moorhouse Pit in 1486.
Flaxley Abbey ..	Cistercian ..	Gloucester ..	Iron working for five centuries in Foresr of Dean.
Fountains Abbey ..	Cistercian ..	Yorks.	12th century, given forges in Nidderdale.
Furness Abbey ..	Cistercian ..	Lancs.	1270, iron ore, and water rights for washing the ore. 1291, 40 forges working.
Hastings Priory ..	Austin Canons	Sussex	Had an iron forge.
Holyrood Abbey ..		Midlothian ..	Colliery at Carriden, nr. Blackness before 1214. 8th century, abbots were noted for iron-work and bell founding.
Jarrow Monastery ..	Benedictine ..	Durham	1313, used coal in monastery.
Jervaulx Abbey ..	Cistercian ..	Yorks.	1281, right to smelt iron. 1333, acquired coal mine.
Kirkstall Abbey ..	Cistercian ..	Yorks.	Worked coal and iron at Ardsley, 1154; Kirkstall, 1200.

Establishment	Order	County	Notes
Kirkstead Abbey ..	Cistercian ..	Lincs.	1161, iron mine and forge at Kimberworth, nr. Sheffield.
Llantwit Major ..		Glam.	Reputed Monastic Mint.
London, Richard, Bishop of			circa. 1160, Treasurer of England. Wrote "Dialogus de Scaccaris" concerning assay of Silver by Cuppellation.
Louth Park Abbey ..	Cistercian ..	Lincs.	12th century, iron bloomery and forge in Derbyshire at Birley (Barlow).
Margam Abbey ..	Cistercian ..	Glam.	1253, lead, coal and iron mining.
Monk Bretton Priory	Cluniac	Yorks.	Worked coal and iron.
Newbattle Abbey ..	Cistercian ..	nr. Edinburgh ..	1210–1214, grant of colliery on sea shore at Preston (Tranent).
Newminster Abbey..	Cistercian ..	Northumberland (Blyth)	1236, seal coal. 1240, coal for iron forge at Stretton, nr. Alnwick.
Nostell Priory	Austin Canons	Yorks.	Early coal miners. By Richard II's time deep pits required drainage. 1372, new Prior made an adit.
Peterboro Abbey ..	Benedictine ..	Northants ..	Saxon Chronicle of the Abbey: 853 records quantities of coal, but some think ref. is to brushwood or peat. (See "Climate and the Energy of Nations," Markham).
Repton Priory ..	Austin nuns ..	Derbyshire ..	835, Abbess leased lead mines at Wirksworth.
Rievaulx Abbey ..	Cistercian ..	Yorks.	1170, worked iron ore in Barnsley (Bilsdale) district.
Robertsbridge Abbey	Cistercian ..	Sussex	Iron and Steel works."
St. Albans Abbey ..	Benedictine ..	Herts.	One of the monks was an artist of great repute in metalwork.
St. Bees	Benedictine ..	Cumberland ..	12th century iron mine at Egremont.
St. David's, Bishop of		Cardigan	1326, lead mine at Llanddewibrefi.
Tintern Abbey ..	Cistercian ..	Monmouth ..	16th century ironworks at Monkswood.
Tynemouth Monastery	Benedictine ..	Northumberland	1330, leased collieries.
Wearmouth Monastery	Benedictine ..	Northumberland	8th century Abbots were skilled iron workers.
Welbeck Abbey ..	Premonstrat ..	Notts	1460, obtained right to make adit in coal mine.
Wenlock Priory ..	Cluniacs	Salop	Iron working. Wm. Corvehill, "a good bell founder," died 1546.
Whalley Abbey ..	Cistercian ..	Lancs.	1529, sea coal used.
York, St. Mary's Abbey	Benedictine ..	Yorks.	William of Towthorpe cast the mortar for the infirmary, 1308.
York, Cell of Lindisfarne	Celtic-Irish ..	Yorks.	9th century monk was skilled iron forger.

APPENDIX B

Patron Saints and Writers

St. Eligius (Eloi), 588–659, goldsmith, native of Dinant, cons. Bishop of Noyon. Patron of metalworkers.

St. Barbara, patroness of miners and metallurgists in Spain, France, Germany.

St. Dunstan, had as pupil at Abingdon, St. Ethelwod.

Theophilus Presbyter (Benedictine monk, 11th century) wrote "Schedula Diversarium Artium." Gives a true picture of technical industry at his time, particularly in the working up of metals, something being also said about their production from ores. He was a skilled worker in metals at Helmershausen Monastery. "An Essay upon Various Arts," 3 Vols. Translated by Robert Hendric, John Murray, London, 1847, pp. 313.

Bartholomew, O.S.F., 1260, wrote of the value of iron.

Albertus Magnus, O.P. (St. Albert the Great). His writings include: "De Rebus Metallicus et Minerailbus." A transmutationist. Recommended juice of radishes and angle worms for surface hardening.

Thomas Aquinas, O.P. (St. Thomas Aquinas) was first to use the word amalgams in his writings. He supported "transmutation" on physical grounds. In his "Summa" discusses physical properties, such as hardness.

Basil Valentine, O.S.B. (15th century, S. Germany). "The Triumphal Chariot of Antimony" was published 1624. In early chemistry gold claimed all attention because of hope of producing elixir of immortality. Otherwise antimony, because of its prolific number of compounds and medicinal properties would have been studied even more. As it was, life-times were often spent on working at it. (See Chemistry and Industry, October 10th and 24th, 1924).

Roger Bacon, O.S.F., Oxford, 1214–1294. Theoretical transmutationist and philosopher.

Fr. Alvaro A. Barba. "El Artos de Metales." 1639. Describes the extraction of metals and discusses physical properties.

Assay of Silver by Cupellation. "De Necessariis Observantiis Scaccarii Dialogus"; commonly called "Dialogus de Scaccaris," by Richard Bishop of London (circa 1160 Treasurer of England). As an assay for silver the cupellation method is generally attributed to the Bishop of Salisbury early in the 12th century. This Bishop of Salisbury was the grand-uncle of Richard, and he appears to have organised the office of Treasurer of England. The Egyptians actually understood the cupellation process. (Roberts-Austen, Cantor Lecture, p. 27).

..Peder Mannson (1460–1534), a monk, published a technological work in Swedish (Engineering, 19/2/43).

The Institute of Metals

Annual General Meeting held in London

THE fortieth annual general meeting of the above Institute was held at the Institution of Civil Engineers, Great George Street, London, S.W.1 on March 16th to 18th inclusive. At the initial meeting on the afternoon of March 16th, the President, Colonel P. G. J. Gueterbock, C.B., D.S.O., M.C., extended a welcome to all members and friends attending the meeting and referred especially to members from overseas. The remainder of the afternoon was given to a discussion of the following papers:—

"Calculation of Loads Involved in Metal Strip Rolling" by Dr. Maurice Cook and Mr. Eustace C. Larke; "Hot-Shortness of Some High-Purity Alloy in the Systems Aluminium–Copper–Silicon and Aluminium–Magnesium-Silicon," by Dr. P. H. Jennings, Dr. A. R. E. Singer and Mr. W. I. Pumphrey; and "A Consideration of the Constitution of Aluminium–Iron–Silicon Alloys and its Relation to Cracking above the Solidus," by Dr. P. H. Jennings and Mr. W. I. Pumphrey. The latter two were discussed jointly. A further paper by Mr. D. McLean on "Straitions: Metallographic Evidence of Slip" was also presented.

The main business meeting was held on the morning of March 17th, when the President briefly presented the Report of Council; the Honorary Treasurer, Mr. H. S. Tasker, presented his report; election of officers for 1948–49 were announced, as was the award of the Platinum Medal for 1948. The new President, Sir Arthur Smout, J.P., was inducted into the Chair and he delivered his Presidential Address.

Award of the Institute's Platinum Medal

The Institute's Platinum Medal for 1948 has been awarded to Mr. Robert Crooks Stanley, Chairman and President of the International Nickel Company of Canada Ltd., in recognition of his outstanding services to the non-ferrous metal industries.

On the organisation of the original International Nickel Company in 1902, Mr. Stanley became Assistant Superintendent of the Camden (N.J.) Works, and subsequently Superintendent. His energy and ingenuity in changing the Camden plant, from an old world chemical manufacturing works of detached units, into an integrated undertaking applying up-to-date metallurgical methods, drew early attention, and in 1904 he was transferred to Bayonne as Assistant General Superintendent of the Orford Copper Company, where again he was responsible for a complete modernisation programme.

"Monel," the white alloy of nickel and copper, owes its discovery in 1905 to Mr. Stanley, who conceived and developed the process for producing the alloy direct from ore without separating the nickel and copper. As General Superintendent of the Orford Works in 1912, Mr. Stanley was in charge of this important source of refined nickel. Under his direction this plant during World War I was successful in substantially stepping up its production to meet war-time demands.

In 1917, he was elected a Director, and in 1918, Vice-President in charge of all operations of the International Nickel Company. When he became President in 1922, on the death of W. A. Bostwick, the nickel industry was at its lowest ebb. One of the initial efforts of Mr. Stanley was the formation of a Development and Research Division, through which thousands of commercial peace-time applications for nickel were established. Thus began probably the most vital phase of growth in the nickel industry. To spur on an increasing demand for nickel, Mr. Stanley arranged for the construction at Huntington, West Virginia, of a large modern rolling mill exclusively devoted to high nickel products; he inaugurated a comprehensive mine and plant expansion in Canada and developed the Frood Mine at Sudbury into one of the world's largest mines.

Following the acquisition of The Mond Nickel Company Ltd., Mr. Stanley continued as President and assumed chairmanship of the Board of Directors on the death of Charles Hayden in 1937.

Again in World War II, Mr. Stanley was responsible for greatly increasing nickel production for its many vital war uses. Soon after the war started, he arranged for extensive expansion of the Company's Canadian mining production, through which a materially increased supply of nickel, copper, and other products was made available to the United Nations in the crucial war years.

Mr. Stanley's achievements in the nickel industry have brought to him many awards including the Thomas Egleston medal, the first gold medal of the Rand Foundation, and the American Society for Metals' medal for the Advancement of Research, while in 1937 the King of Belgium conferred upon him the Order of Leopold. In September, 1947, Mr. Stanley was awarded the "King's Medal for Service in the Cause of Freedom." He has also been awarded the degrees of Sc.D. of Columbia University and the D. Eng. degree of the Stevens Institute of Technology and of the Rensselaer Polytechnic Institute.

Presidential Address

In his opening remarks following his induction as President of the Institute Sir Arthur Smout said he had been called upon to succeed one of the most successful of a long line of successful Presidents—one who has worked hard for the Institute, especially during the past two difficult years of reconstruction and reorganisation—and who has graced the chair with charm and dignity.

In this Address, he thought it desirable and opportune to re-examine the reasons which led to the establishment of the Institute, the ideals its founders had in mind, and the principal objects which they intended the Institute to fulfil, and to consider how far these ideals and objectives had been achieved.

Objects and Early History of the Institute

The Institute of Metals was founded forty years ago, after lengthy deliberations. The idea came from a young engineer, Walter H. A. Robertson, who on January 20th, 1908, directed the attention of the editor of *Engineering* to the desire of progressive manufacturers "for an Institution of a similar kind to that valuable organisation possessed by the Iron and Steel Traders." The idea was quickly taken up and Mr. W. H. A. Robertson—who was present at this meeting—can justly claim to be the initiator of the movement which led to the Institute's formation.

A preliminary meeting of those interested was held in Manchester on March 10th, 1908, but those present were very cautious and decided to think about a possible association, and it was not until June 10th, 1908, at a meeting held in London, that the all-important decision was taken. Several titles for the new organisation were suggested, but the title finally accepted, which the Institute now holds, is exceedingly well chosen.

There appears to have been a fear present in the minds of all the speakers at Manchester that the new Institute might fail through the supposed disinclination of the all-powerful manufacturers to allow their technical people to contribute papers or to take part in the discussions. We were indeed "closed shops" in those days. And the man who advocated caution and further consideration was Rosenhain—of all men! Indeed, he framed and moved the resolution advocating the desirability of a later meeting before a definite constitution was framed. It is interesting that neither Robertson—who gave the first impetus to the movement—nor Rosenhain—the mover of the resolution—were members of the first Council.

In addition to Sir William White, whose Presidential Address was a masterpiece of clear, concise English, setting forth the objects of the Institute and its ideals, there were among others on the first Council such men as Norman Cookson, Gowland, Gerard Muntz, Engineer Vice-Admiral Oram, Thomas Turner, Boeddicker, Tom Bayliss, Carpenter, Nisbett and Dr. R. S. Hutton, and we should not overlook the pioneer work of W. H. Johnson of Manchester, the first Honorary Secretary. Alas, all have passed over with the exception of that pioneer scientific industrial metallurgy, Thomas Turner and R. S. Hutton. Reference should also be made to Shaw Scott, Birmingham's first graduate in metallurgy, who was the first secretary and editor of the Institute, to whose guidance and devotion it owes much.

It is well to recall those days and to pay tribute to those pioneers at this the fortieth annual Meeting of the Institute.

Sir Arthur referred briefly to the opening meeting, held in Birmingham in November 1908 and to the range of subjects discussed at that meeting which showed very clearly the yawning chasm of ignorance and prejudice the Institute had to bridge in order to bring scientists and practical men engaged in works operations into close contact. In having done, and in continuing to do, this, the Institute has probably registered its most important achievement over the years of its existence. But the past is useful insofar as it provides a lesson and a stepping-stone for the future and the question arises: What is the role of the Institute of Metals; what part can it play in the development of the art and science of metallurgy? What contribution can it make towards the well-being of mankind—towards the building of a better Britain?

The Future Role of the Institute

Progress in all branches of learning is largely dependent on the full interchange of knowledge among workers pursuing similar lines of study. Metallurgy is an old art and a new science. It is the duty of those of us engaged on the industrial side to make a continually improved product at a continually reduced cost of production available to the user and, through him, to the general public, passing on to the ultimate consumer most of the benefits to be derived thereby. This is the means by which the standard of living can be permanently raised. The important factor in achieving this is "continuity of production," and there is a temptation to contrast works processes as carried out in the chemical industry with those in the metallurgical field. The great chemical industry is essentially based, on continuous processes operating 365 days a year. 24 hours a day. The manufacturing metallurgist and the user work, in the main, on batch processes, nowadays operating a five-day week; no wonder capital investment per ton/year plays so large a part in our considerations. Up to now we have striven to combat this by speeding up our motors, rolling mills, wire drawing machines, presses, etc., and as metallurgists we have solved the problem of materials which will stand up to these increased demands; but Sir Arthur suggested this was merely toying with the problem.

The best method of increasing production and obtaining the maximum return from the capital employed is the decrease, or the complete elimination, of "idle time" rather than in trying to increase speed *per se*. It is self-evident that if our metallurgical plants must start, operate, and stop, to produce a product, not only will the efficiency of the plant be increased by eliminating the starting, stopping, and stand-still time, but the resultant product will be greatly improved if it is produced by a continuously uniform operation. Its cost too, will be lessened even if the actual speed of the machine when operating continuously is lower than the speed of the machine in the first instance. This question of elimination of idle time, this all-important matter of continuity of operations is a major lesson we in the metals industry can learn from the industrial chemist.

Admittedly, the industrial metallurgist can point with pride to the improvements made in production processes along the lines of eliminating waste or idle time, to continuous methods of casting, to the so-called continuous rolling on tandem mills, and to continuous wire-drawing technique, etc. All of these are developments of recent years, but the fact remains that continuity of operations is not a strong feature of the metals industry generally. We are, by and large, a "batch process" industry, and to any industrial metallurgist laying out a new plant, the technical approach of the industrial chemist is worthy of close consideration.

The metallurgy industry is as old as Tubal Cain himself, whereas as Hume-Rothery has pointed out "much of the chemical industry of to-day is a large-scale development of previous laboratory research and the industry has developed *after* the main principles of chemistry have been established." On the other hand, we metallurgists always seem to be trying to explain things which have been practised over many years; we are attempting to superimpose a science on an old established art. This gives rise to its own peculiar and special problems, both in the field of research and in works practice. And again to quote Hume-Rothery: "The development of physical metallurgy has been very different, since here, traditional practices have been handed down through the ages and it is only in comparatively recent times that scientific methods have been used. It should be appreciated that, scientifically, there is nothing more improbable in the idea of building up an alloy of desired properties from a knowledge of the characteristics of the different kinds of atoms, than there is in building up synthetic dyestuffs with desired colours. The difference between the two problems lies, largely, in the fact that whereas the dyestuff chemist works with a soundly established science behind him, in physical metallurgy the general principles are only just being revealed."

Metallurgy too, is not without its parallels. Most of our modern non-ferrous alloys, many of which played so important a part in the war effort, have been built up deliberately, not as the result of numerous trials and errors as in the past, but on general scientific principles well established by investigators in the so-called academic field. The aluminium light alloy which formed the basis of our aircraft construction is a practical instance, and many others could be cited did time permit.

The progress of all science is essentially a cumulative one; we build our work on the foundations established by our predecessors. This Institute is a learned society; it is not a research association. We exist to collect facts, to exchange ideas, to disseminate and record new knowledge, and to build up works technique and user practice on the information and experience thus acquired. It is all the more important, therefore, that those of us who are concerned with the practical application of metallurgy should encourage on every hand those of our colleagues who are engaged in seeking new knowledge and should see that every possible step which lies within our power is taken speedily and scientifically to adapt this knowledge to industry.

The Responsibilities of Members

The Non-Ferrous Metals Industry, for which this Institute stands as the accredited learned society, covers a wide field; but fortunately the knowledge acquired, collected, and disseminated by the Institute is capable of application over an equally wide, if not wider field. Thus, our meetings, discussions and publications are of material interest to all members, regardless of the particular product they manufacture or use, or the pet scientific problem they have under review at the moment. The value of the Institute's work is perhaps greatest to those user and manufacturing members who, either by virtue of their own knowledge or the knowledge of technical men in their employment, are most capable

of understanding it and of grasping quickly and clearly its implications and significance in relation to the day-to-day problems facing the industry on the production side.

The ordinary member should resist the temptation to say "I am not interested in the manner in which different kinds of atoms occupy a common lattice." Let so-called practical men beware of such a narrow and detached attitude, for herein may be the solution of a most pressing problem. If the language of the scientists is beyond our comprehension, it is up to us to see that as industrialists we have on our staffs technical men with wide vision and great adaptability who have been trained to grasp the full significance of such observations. We must give them facilities for keeping in touch with the latest research, even of the most obstruse kind, and enable them to interpret it to works practice for us.

The great appeal of the Institute of Metals to me lies in the fact that it supplies such an important link between higher management engaged in the problems connected with our industry, and the scientist and technician searching for fundamental knowledge and its applications.

Technical Sessions

The afternoon of March 17th and the morning of March 18th were given to the discussion of technical papers including "The Effect of Single- and Multi-Hole Die Extrusion on the Properties of Extruded Aluminium Alloy Bar," by Dr. L. Northcott, Mr. D. McLean and Mr. O. R. J. Lee; "The Young's Modulus of Some Aluminium Alloys," by Dr. Ing. N. Dudzinski, Miss J. R. Murray, Mr. B. W. Mott and Dr. B. Chalmers; "Pressure and Creep Tests at Constant Hoop Stress on Lead and Alloy 'E' Pipes," by Mr. A. Latin; and "Recent Developments in Corrosion Resistant Aluminium-Magnesium Alloys" by Dr. P. Brenner and Dr. W. Roth. These papers, together with those presented on March 16th, are briefly outlined in the following notes:—

Works Visits

The afternoon of the concluding day was given to works visits. These visits to works was a prominent feature of the annual meeting before the war and one which was appreciated very much, it is not surprising, therefore, that many members looked forward to them on this occasion. The only difficulty was that they were all organised for one afternoon, thus, it was only possible to make one visit and the choice was not easy.

The works visited included: Aluminium Plant and Vessel Co. Ltd.; Enfield Rolling Mills Ltd.; Enfield Cables Ltd.; High Duty Alloys Ltd.; both Rainham and Waltham Cross works of Murex Ltd.; and J. Stone and Co. Ltd.

CALCULATION OF LOAD INVOLVED IN METAL STRIP ROLLING

By Maurice Cook, D.Sc., Ph.D. and Eustace C. Larke

SEVERAL attempts have been made in recent years to derive a general formula for calculating the load involved in the rolling of strip or sheet and several formulæ have been advanced, but, because of several assumptions they contain, they are only of limited application and only very approximate. Moreover, in applying the formulæ, arbitrarily chosen values have to be adopted for the coefficient associated with the friction conditions in the roll throat, and it is necessary to determine experimentally the resistance to deformation as a function of the amount of reduction in thickness effected. Furthermore, none of the methods so far proposed makes any allowance for the effect of roll-face distortion.

The most comprehensive and exact analysis of the dynamics of rolling has been carried out by Orowan,[1] who did not assume that resistance to deformation was constant, but took into consideration work-hardening which takes place during cold rolling. In using this method of calculation, however, it is necessary to determine experimentally curves of resistance to deformation, and this may necessitate consideration of the effect of inhomogeneous deformation. In view of the doubts and difficulties associated with the determination of acceptably accurate values assignable to the influence of roll flattening, friction, and resistance to deformation, the authors have given attention to the development of a method of rolling-load computation which avoids the necessity of determining values of the various factors individually, by any theoretical or practical procedures. The method is based on, and makes use of, a minimum of data in the form of initial rolling-load measurements. It has been developed on the assumption that the magnitude of the pure work of rolling is independent of the number of passes used in effecting a given reduction in thickness, and the basic experimental data required consist only of a few measured values of the rolling loads developed in rolling one material under a series of different sets of rolling conditions. From these data, rolling loads can be readily computed for practices involving any number of passes, or a sequence of reductions in thickness can be established for which a constant load is developed in each pass.

Results calculated by the method can be directed checked against measured values, and comparisons which have been made between calculated values and those obtained experimentally on steel and copper, in the course of investigations carried out by the British Iron and Steel Research Association, show good agreement over the range of reductions encountered in normal rolling mill procedure.

HOT-SHORTNESS OF SOME HIGH-PURITY ALLOYS IN THE SYSTEMS ALUMINIUM-COPPER-SILICON AND ALUMINIUM-MAGNESIUM-SILICON

By P. H. Jennings, Ph.D., B.Sc., A. R. E. Singer, Ph.D., B.Sc., and W. I. Pumphrey, M.Sc.

THE work described in this paper was carried out on similar lines to a previous investigation of the hot-shortness of aluminium-iron-silicon alloys,[2] but it deals with a more extensive range of alloys. The results cover the range copper 0–10 and silicon 0–4% in the aluminium-copper-silicon system, and magnesium 0–10 and silicon 0–5% in the aluminium-magnesium-silicon system.

The investigation was carried out by means of a ring-casting test and a restrained-weld test. The first alloying additions to pure aluminium cause a rapid increase in hot-shortness which, after passing through a maximum, gradually decreases as the alloy content is raised.

In the aluminium-copper-silicon system, additions of silicon are more effective than additions of copper in reducing hot-shortness once the maximum hot-shortness has been passed. In the aluminium-magnesium-silicon system, the most-hot-short alloys are those near the quasi-binary aluminium-magnesium silicide line. The presence of silicon or magnesium in excess of the Mg_2Si ratio causes, in general, a reduction in cracking.

1 E. Orowan, *Proc. Inst. Mech. Engrs.* 1943. **150**, 140
2 Singer, A. R. E., and Jennings, P. H., *J. Inst. Metals*, 1947, **73**, 273.

The hot-shortness of binary and ternary alloys appears to depend upon the extent of the hot-short temperature range, i.e., that range in which the alloys possess appreciable strength but no elongation; this in turn is a function of the freezing range and the proportion of liquid freezing at a constant temperature. In ternary systems, however, other factors, such as the temperature at which secondary separation begins, may be of importance. The application of the theory of hot-shortness to aluminium-magnesium-silicon alloys is complicated by the magnesium silicide phase.

Tests show that good mechanical properties are possessed by those aluminium-copper-silicon alloys which are likely to be of use from the point of view of hot-shortness.

Several industrial alloys lie within the ternary fields investigated, the results, therefore, have a direct practical application. Alloys containing approximately 3% copper and 4% silicon, for instance, have good casting properties. More recently, similar alloys have been prepared in the wrought form, and it has been found that they can be welded into complex structures without danger of cracking. The alloys of this type so far used in industry have been confined to limited areas of the aluminium-copper-silicon ternary diagram, and the present work enables some estimate to be made of the suitability of other alloys for casting and welding.

In the aluminium-magnesium-silicon system, there are two main types of industrial alloys in relation to which the results of the present work are likely to be of interest. The first type includes the alloys of aluminium with 3–7% magnesium, which sometimes weld unsatisfactorily on account of gas porosity. To remedy this tendency silicon is sometimes added to the extent of 0·5–1·0%. The likely effect of this addition on the hot-shortness and tendency to weld cracking can deduced from the results of this investigation. The second type comprises the heat treatable alloys which depend for their properties on the precipitation of magnesium silicide. These alloys are particularly prone to hot cracking in both castings and welds, and the possibility exists of minimising this defect by slight alterations in composition.

A CONSIDERATION OF THE CONSTITUTION OF ALUMINIUM-IRON-SILICON ALLOYS AND ITS RELATION TO CRACKING ABOVE THE SOLIDUS

By P. H. Jennings, Ph.D., and W. I. Pumphrey, M.Sc.

IN an earlier paper,[1] a theory was advanced relating the results of the experimental work on the aluminium-silicon alloys to their constitution, and it was suggested that this theory might be generally applicable to the cracking of aluminium alloys at temperatures above the solidus. At the time of publication of the results for aluminium-iron-silicon[2] alloys, the correlation with the constitutional diagram was, for a variety of reasons, far from complete; but after a more exhaustive study of the literature relating to equilibrium and metastable conditions in this system, the position has become considerably clearer.

While the constitution of aluminium-silicon alloys is simple and is known with accuracy that of aluminium-iron-silicon alloys is of much greater complexity; and, although it has been the subject of numerous investigations, several points remain to be clarified. For this reason, it was thought unsatisfactory to select any single work on which to base the discussion of the hot-shortness, and the conclusions of the more comprehensive researches have been compared.

It is concluded that the relationship between the constitution and the susceptibility to cracking of aluminium-rich aluminium-iron-silicon alloys appears to be similar to that suggested for binary aluminium-silicon alloys. This relationship is, however, less easily established in the ternary system because of the uncertainty regarding particular features of the constitutional diagram.

The small tendency to cracking of alloys in which there is a preponderance of iron over silicon is apparently due to the fact that, as the iron content increases, the ternary-eutectic arrest at 577° C. diminishes in magnitude and becomes unobservable at an iron : silicon ratio of about 1 : 1. The major part of the solidification is thus completed at about 615° C., and there is an effective shortening of the liquidus-solidus interval. This contention has been supported by the microscopic evidence of other investigators, which provides the additional information that when the total silicon content is less than the iron content, the free silicon is present in a form which renders it ineffective in promoting a tendency to cracking at temperatures above the solidus.

1 Singer, A. R. E., and Jennings, P. H., *J. Inst. Metals*, 1946, **73**, 197.
2 Singer, A. R. E., and Jennings, P. H., *J. Inst. Metals*, 1947, **73**, 273.

STRIATIONS: METALLOGRAPHIC EVIDENCE OF SLIP

By D. McLean, B.Sc.

IT is known that slip lines can be seen on polished surfaces of a deformed metal if the metal is polished before deformation. Strain lines can also be seen on etched sections prepared after deformation when the deformation exceeds about 20%; the relation between strain lines and slip lines is uncertain. It seems to be believed, however, that no evidence of slip or, indeed, of any microstructural change, apart from slight elongation of the crystals, can be seen in a lightly deformed metal on a section polished and etched after deformation. It is shown in the present paper that this belief is incorrect since markings which are evidence of slipped planes can be seen in brass when a suitable polishing and etching technique is adopted.

THE EFFECT OF SINGLE- AND MULTI-HOLE DIE EXTRUSION ON THE PROPERTIES OF EXTRUDED ALUMINIUM ALLOY BAR

By L. Northcott, D.Sc., D. McLean, B.Sc., and O. R. J. Lee, M.Sc.

IN the mass production of a conical component machined from extruded aluminium alloy bar in BA35 and D.T.D.423A alloys, numbers fell under suspicion owing to them having a fan-shaped streaky appearance. It was feared that the streakiness might indicate low circumferential properties and this was supported by preliminary tests. The cylindrical bars from which the components were made, were extruded through single- or multi-hole die plates, and multi-hole die extrusion was suspected as being responsible for the streakiness and circumferential weakness, which in some components was very marked. It thus became clear that the original problem was part of the general question of the effect of extrusion through different types of die plates. An investigation was therefore initiated into the effect of extrusion, through die plates having 1, 2, 3 or 4 holes, on the mechanical properties of the bars. The experimental work showed that in many instances there was a variation in properties along the length of the bar; consequently, most tests were repeated many times (as an extreme instance nearly 150 drift tests

were carried out), but only mean results are given in the present paper, and the question of scatter is dealt with separately, since it assisted in deciding the cause of the variable properties.

Billets of the two alloys were extruded through die plates having 1, 2, 3 or 4 holes, all the multi-holes being symmetrically disposed about the centre. Transverse sections of bars extruded through multi-hole die plates showed a structure termed " radial flow," converging to that portion of the surface marked by the streak ; this structure was absent from bars extruded through single-hole die plates. Segregation of particle constituents was found near the periphery in the zone of radial flow and, consequently, the circumferential tensile properties of multi-hole material were inferior to those of single-hole bar. Small-scale experiments with lead-bismuth composite and coloured plasticine showed that the segregate originated from the axial zone of the billet ; with a die plate having a single central hole, on the other hand, the axial segregate was confined to the axis of the single bar, where it is relatively harmless. Provision of a central hole in 3- and 4-hole die plates confined the segregate to the axis of the central bar, and this advantage has been confirmed by industrial trials.

THE YOUNG'S MODULUS OF SOME ALUMINIUM ALLOYS

By N. Dudzinski, Ing. Chem., Miss J. R. Murray, B.A., B. W. Mott, M.A., and B. Chalmers, Ph.D., D.Sc.

THE development of high-strength aluminium alloys, combined with certain trends in aircraft design, has led to a situation in which it is envisaged that the failure of a structure may be brought about by elastic instability of compression members before the tension members fail in tension. The onset of elastic instability is governed, other things being equal, by the Young's modulus of the material concerned ; consequently, considerable attention has recently been paid to the possibility of improving aluminium alloys in this respect.

Certain experimental alloys based on the aluminium-silicon eutectic gave abnormally high values of Young's modulus. Statistical analysis of results available indicated that high Young's modulus was related to the composition ; in particular to the amounts of manganese and nickel in the alloys. It was decided, therefore, to investigate the effects of varying alloying elements, such as silicon, manganese, nickel, cobalt and beryllium, on the Young's modulus. The work described in this paper was undertaken in order to arrive at an understanding of these effects and to provide the basic information that is required for the development of alloys combining a high value of E with acceptable tensile and other properties.

Various binary, ternary and complex aluminium-base alloys were prepared with this object. All the five elements investigated were found to enhance the value of E, manganese having the greatest effect (about $0{\cdot}34 = 10^6$ lb./in.^{2} for 1 wt.-%) and the elements beryllium, cobalt, nickel, and silicon having decreasing effect in that order. Beryllium and cobalt both have a greater effect in the presence of 12% silicon than in the respective binary alloys, but the effect of beryllium is smaller in alloys containing 2·5% copper. Attempts to relate these effects with the constitution of the alloys have met with partial success. The values of E obtained on forged and heat-treated aluminium-nickel and aluminium-silicon alloys were in agreement with those given by corresponding alloys in the chill-cast condition.

On a complex aluminium alloy containing silicon 12, copper 2, nickel 2, manganese 1, magnesium 0·6, cobalt 0·2, and titanium 0·05% (with the exception of sand-cast and thin sheet materials, which can be explained), the value of E was the same, within experimental error, for materials in the cast, forged, and rolled conditions, both before and after heat-treatment. Complex alloys containing varying amounts of nickel and manganese gave higher values of E than those obtained by calculation from the previously-determined individual effects of the various elements.

Further developments of the work are discussed briefly, including the possibilities of developing alloys combining high strength with improved Young's modulus. Although no effort was directed towards this end, the indications are that, by suitable modification of composition, alloys can be produced having these tensile properties : 0·1% proof stress, 21 tons/in.^{2} ; maximum tensile stress, 28 tons/in.^{2} ; elongation on $4\sqrt{A}$, 4% ; Young's modulus, 12×10^6 lb./in.^{2} It is considered that it will be possible, as a result of further work, to produce alloys with a Young's modulus greater than 12·0 without any marked lowering in these tensile properties.

An Appendix describes tests designed to determine whether, for aluminium alloys, the Young's modulus in tension is the same as the Young's modulus in compression. Two materials were used : one in the form of bar to British Standard Specification No. 6L1, and the other in the form of sheet to Specification D.T.D. 646A. The results indicated that, within the limits of experimental error, the moduli in tension and compression were the same.

PRESSURE AND CREEP TESTS AT CONSTANT HOOP STRESS ON LEAD AND ALLOY "E" PIPES

By A. Latin, Ph.D., M.Eng.

TESTS have been carried out and are still in progress at the laboratories of British Insulated Callender's Cables Ltd., on lead and lead alloy pipes of cable sheath size, to determine the extension obtained before fracture at different rates of creep under internal pressure. The pressures used have ranged from a few to the order of 150 lb./in.^{2} (the latter for short-time tests with the harder alloys), corresponding to hoop stresses ranging from about 100 to 1,500 lb./in.^{2}, and each test has been carried out under conditions of constant pressure. This leads to approximately constant creep rate over most of the test period, and is analogous, although not exactly so, to constant-load creep testing, in the ordinary way, on specimens of strip, &c.

These tests are also analogous to constant-load creep tests in that the applied stress increases as creep progresses. The hoop stress f, produced in the wall of a pipe of internal diameter d_1 and thickness b by internal pressure P is, using the formula for thin-walled pipe given by : $f = Pd_1/2b$. As creep progresses, d_1 increases and b decreases ; consequently, f increases if the pressure is constant.

The advantages of creep tests under constant-stress conditions has been pointed out by Andrade[1 2 3] and he has derived the following expression for creep rate under such conditions —

$$l_s = l_o\,(1 + \tfrac{1}{3}\beta t^{1/3})e^{kt},$$

1 Andrade, E. N. da C., *Proc. Roy. Soc.*, 1911, [A], **84**, 1.
2 Andrade, E. N. da C., *Proc. Roy. Soc.*, 1914, [A], **90**, 329.
3 *Nature*, 1946, **157**, (3989), 469.

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Precision Castings in High-Melting Point Alloys for General Engineering Purposes

By Frank Hudson, F.I.M.

Castings in high melting point alloys, possessing high dimensional accuracy, are being produced by the precision investment casting process to overcome forging and machining difficulties of other methods, but the production costs are high. In the present paper, read before a recent meeting of the Midland Societies, the author reviews those factors which have a bearing on economic production methods and discusses possible new applications for such castings. He is confident that there is a place in engineering for the process, but that future development will depend on close co-operation between engineer and founder.

IN a recent editorial[1] on the subject of precision castings, it was suggested that, at the moment, "nothing adds up." Those with an intimate experience of the problems connected with the production of precision castings may not altogether agree with the full text of this editorial, but will appreciate the need for a better understanding on the economic use of such castings in industry. There is a distinct tendency for engineers and founders to base the future value of precision castings upon existing applications, and this is giving rise to distorted viewpoints and wrong perspectives.

Most of the work done so far in both the United States of America and in Great Britain, relative to precision castings, has been in connection with gas turbine developments, as applied to aeronautical engineering. This has entailed the production of castings to a very high standard of quality in alloys difficult to handle in the foundry. Every casting produced must be 100% sound, and this is ensured by most exacting inspection methods. It is doubtful whether the yield of usable castings has ever exceeded 50% of the total manufactured. A yield of 30%, in view of the conditions imposed, might even be considered satisfactory. As a result, production costs are abnormally high and foundries making precision castings of the type at present in demand are gradually coming to the decision that the production of such castings is not a commercial proposition, unless the engineer is prepared to pay a good price for them. It is, however, realised that the future economic development of the gas turbine or jet engine may well depend upon the ability of the founder to produce these specialised castings at a much lower cost than is possible to-day, and therefore it is essential to maintain research and development work in this direction. From the economic point of view this would be greatly facilitated if precision castings could be extended into the field of general engineering where requirements, such as dimensional tolerances and quality, etc., are less severe. In other words, it is now essential to develop "bread and butter" business to help spread the high costs incurred in the development of the more specialised applications. It would appear that founders in the U.S.A. are already working on similar lines.

The purpose of this paper, therefore, is to review those factors and requirements which have a bearing on economic production methods and to discuss possible new applications for precision castings in high-melting point alloys of the type mentioned.

1 *Foundry Trade Journal*, 1947, LXXXII, 199.

The Utility of Precision Castings in Engineering

The application of precision casting methods to aeronautical engineering purposes was developed in the early war years and has since become of increasing interest for the production of small engineering parts, which, for economic or technical reasons, do not lend themselves to machining, or for which adequate machining facilities are not available, but in which dimensional accuracy and surface-smoothness are essential. Alternatively, precision cast parts can be utilised to eliminate or reduce costly machining operations and provide, in many instances, an alternative to die-castings in high-melting point metals and alloys such as steel, cast iron, nickel alloys, etc., which cannot normally be die-cast. The process can also be economically applied to small runs of castings or stampings in a wide variety of ferrous and non-ferrous alloys where die costs are too expensive.

Precision Casting Methods at Present Employed

All of the precision castings so far produced in the U.S.A. and in Great Britain have been made by what is known as the "lost-wax" process. During the last few years, a considerable amount of information has been published in the technical Press on production methods and these can be broadly divided into two groups according to whether the castings are required in high- or low-melting point alloys.

The technique employed for high-melting point alloys, such as steel, nickel alloys, cast iron, etc., has been ably outlined in a recent paper by H. Evans, P. S. Cotton and J. Thexton[2] from which it will be found that the process requires ten production stages :—

1. Preparation of a master-pattern in an easily machinable material, such as wood, steel or brass, to the design of the component of which it is required to prepare numerous castings, but made oversize on all dimensions (to an extent arrived at by experiment, but often around 2%) in order to allow for shrinkage during the latter stages of the process.

2. Preparation of a split master-die, from the master pattern, in a low melting-point alloy, or in rubber. Where the component is simple in shape it may be unnecessary to use the above stages. Instead it may be possible to prepare the split die in steel or brass by normal machining operations.

3. Preparation of wax patterns, by injecting wax into the master-die. The necessary wax patterns of gates and risers, to enable adequate feeding of the

2 *Foundry Trade Journal*, 1947, LXXXII, 205–210, 223–227, Disc., 337–342.

castings, are made separately in a similar way or by pouring molten wax into moulds having cavities of the desired shapes.

4. Joining wax gates and risers to wax patterns by the use of a heated spatula, and attachment of the inverted wax assemblies to a waxed base plate.

5. Application of a refractory coating by dipping or by spraying with a finely crushed refractory suspended in a liquid binder.

6. Investment of coated assemblies in a container in a refractory suspended in a liquid binder. The container is vibrated.

7. Drying of invested assemblies, at a temperature below the " melting point " of the wax.

8. Melting-out wax patterns from the invested assemblies followed by firing the moulds by heating slowly to a temperature which is dependent upon the intricacy of the casting to be made and on the pouring temperature of the alloy employed.

9. Pouring the molten alloy from the melting furnace into the cavity of the hot mould.

10. Extraction of the casting, when cold, from the mould, and removal of the gates and risers, followed by cleaning the surface of the casting by shot-blasting.

In passing, it might be mentioned that the production of precision castings in alloys of lower melting point, such as brass, bronze and light alloys, with the exception of magnesium, is less complicated. The application of a refractory coating to the wax pattern, as mentioned under stage 5 above, is unnecessary and satisfactory results are obtained by simply investing the pattern direct with a relatively cheap plaster of paris or gypsum mixture. Furthermore, drying costs of moulds made in the latter materials are much less than those used for high-melting point alloys and firing of the moulds after drying is usually omitted. In the case of magnesium alloys, however, these react with plaster base investments and consequently similar production methods to those outlined for high-melting point alloys must be employed.

Factors Affecting Economic Production

(*a*) Limitations of present production methods.

Production methods at present entail the use of expensive materials, take some time to complete and have to be done by hand. Furthermore, as yet, little attention has been given to the possibility of reclaiming the refractories employed. It is highly probable that production costs would be reduced if the process could be mechanised and an endeavour made to decrease production time and the cost of raw materials.

It is customary, for example, in the process used to-day, to spray the wax pattern with a fine-grained refractory. This sprayed coating must be allowed to air-dry for some considerable time, possibly up to 12 hours. It may even be necessary to apply a second coating which will increase the operational time still further. Then the sprayed and air-dried patterns are invested. During the latter operation the invested mould is vibrated for periods up to 2 hours in order to consolidate the investment and to remove air bubbles. After this the mould is subjected to a long drying and firing schedule :—

1. A preliminary drying period of up to 48 hours at 35°–45° C. to remove moisture. If this initial drying is unduly accelerated there is a tendency for the invested mould to crack.

2. After drying the moulds are heated to approximately 200° C. to melt out the wax pattern. Two hours are usually required for this operation.

3. The moulds are next transferred to a furnace and slowly heated to a temperature of between 850°–1,000° C. This treatment entirely removes all the remaining wax and brings the moulds to the temperature at which they are to be cast. This operation requires at least 8 hours.

Altogether up to 72 hours may be required after the wax pattern has been sprayed before moulds are available for casting. Furthermore, it is doubtful, in view of the nature of the operation involved, whether this time could be effectively accelerated by mechanisation.

There is, undoubtedly, considerable scope for effecting a reduction in production costs. Consideration might be given to the development of a single moulding composition to replace the existing types of pattern spray and investment mixtures. This moulding composition should contain the minimum amount of binder so that moulds can be made automatically on a press and dried and fired in a fraction of the time it takes to-day. The moulding composition must be suitable for use with any metal and capable of reclamation. Graphite, or some other form of carbon, could be usefully considered for this purpose. As an experiment some years ago, a mould for casting turbine blades was machined from a block of graphite and the castings produced were equal or better than those precision-cast by existing methods. Furthermore, a graphite mould ensures more rapid solidification of the metal which may be of great assistance in obtaining maximum soundness and strength in certain designs of castings. It should not be difficult to evolve a suitable moulding composition of the type suggested.

From a commercial point of view, a Frenchman, by the name of Carré, appears to be the first to have had conspicuous success in producing a carbonaceous mixture suitable for moulding or extrusion methods. When Carré studied the problem, the Gramme dynamo had just appeared and a host of inventors were struggling to produce good arc lamp carbons. Carré's carbon mixture, which was patented in 1876, was as follows :—

Very pure coke, finely pulverised	15 parts
Calcined lampblack	5 parts
Syrup of sugar	7 to 8 parts

This was mixed into a stiff paste and extruded through dies on a hydraulic press working at a pressure around 1,600 lb./sq. in. The carbon rods, so formed, were then baked and fired at approximately 1,000° C. It is interesting to note that even to-day the general operations in the manufacture of carbon electrodes have remained practically the same.

In 1899, O. G. Pritchard, an Englishman, published a series of articles on the manufacture of carbon electrodes. His raw material was Ceylon graphite. Pritchard used cane sugar as a binder, explaining that he had failed in attempting to use such substances as tar oils and other hydrocarbons, gums, etc., for the reason that all these materials produced undue porosity after the carbons were baked. Whilst this porosity may be detrimental to a product such as an arc-lamp carbon or electric furnace electrode, one might imagine that it may be an actual advantage in a carbon mould used for precision-casting purposes.

To-day, coal tar or pitch are the binders most commonly employed for the production of carbon articles.

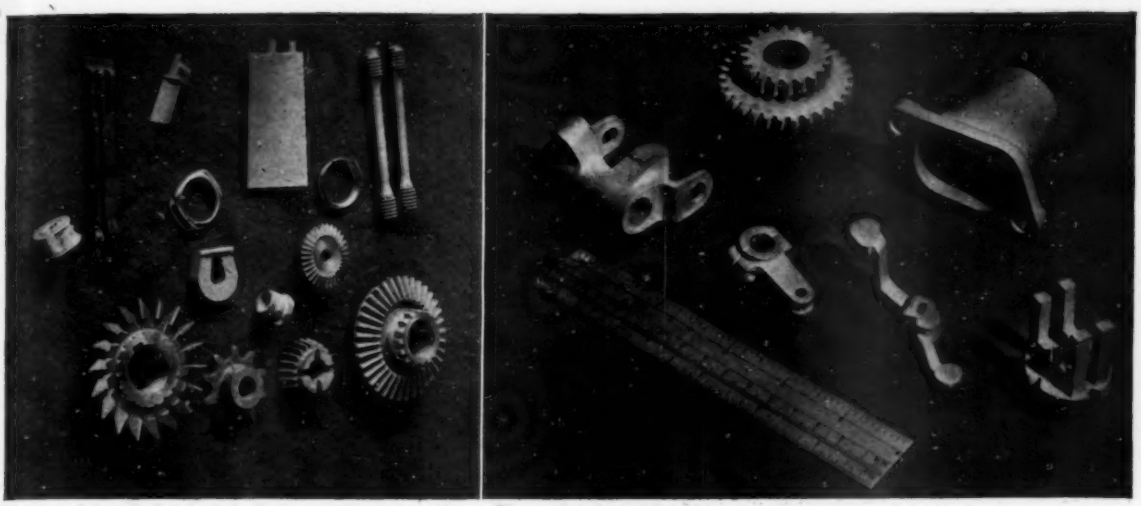


Fig. 2.—Group of miscellaneous articles suitable for precision casting.

Sprocket wheel and gate for cine-projector. Gas turbine blades. Test-bars. Watch case. Magnet. Screwed packing gland. Gear and sprocket wheels. Milling cutter. Precision-casting methods can be utilised to exploit the wear-resisting properties of very hard alloys which cannot be commercially machined.

Should a carbon-base moulding composition be successfully developed for the production of machine-made moulds, it may, of course, be necessary to utilise a stronger material than wax for patterns. At present, a considerable amount of thought is being given to this problem as trouble has already arisen from the sagging and distortion of wax patterns on hot days. This trouble has been particularly common in America and the latest developments in pattern materials include the use of plastics and fusible alloys.

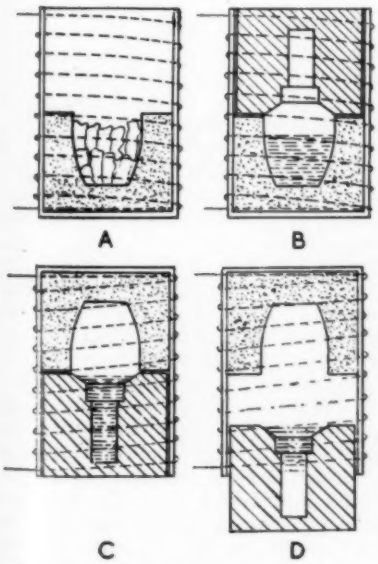


Fig. 1.—Method for controlling solidification by induction heating.

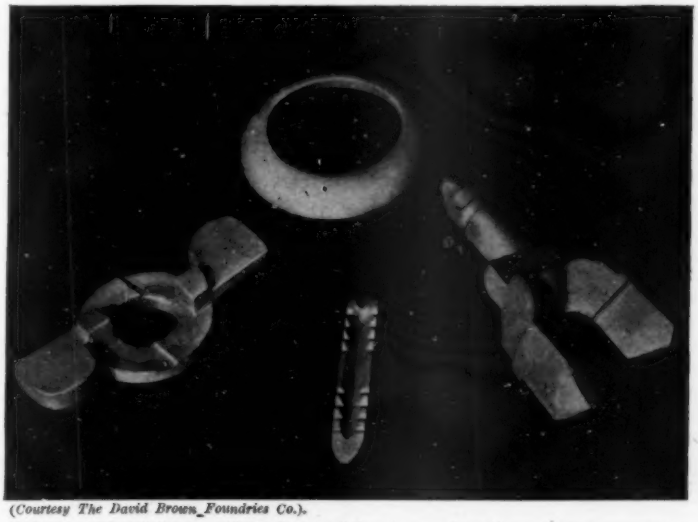
(Courtesy The International Nickel Co., Inc.).

Fig. 3.—Precision castings in high-melting point alloys for general engineering purposes, made in the U.S.A.

Monel yoke, lever, lever with " D " hole, actuator, double gear and stainless steel manifold fitting.

(*b*) Effect of the engineer's requirements on economic production (e.g., design, dimensional tolerance, inspection methods, etc.).

It is most important that careful consideration be given to the design of precision castings, particularly if these are required for high-duty service and subject to exacting inspection methods. So far as turbine blade castings are concerned, it is essential that sufficient taper be provided along the length of the blade to ensure directional solidification from the tip to the root. When castings have to be made in certain high melting-point alloys with a short freezing range, and when the section of the metal is on the thin side, very often even sufficient



(Courtesy The David Brown Foundries Co.).

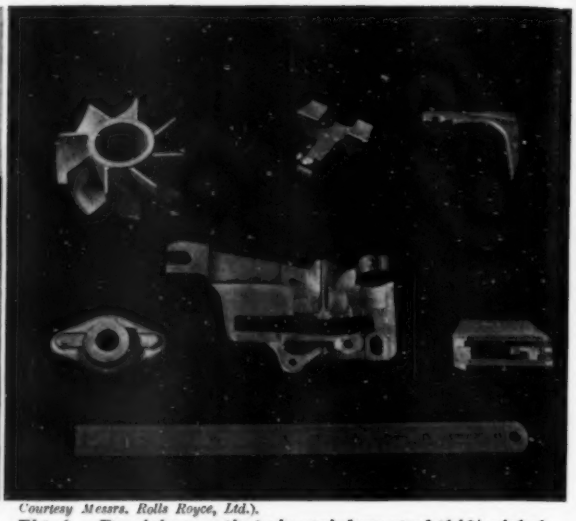
Fig. 4.—Precision castings in steel for general engineering purposes.

Alloy steel piston sealing ring heat-treated to give a tensile strength of 60–70 tons/sq. in; Mild steel jaw for joiner's brace ; mild steel cast drill bit. The ends of this bit are subsequently tipped with hard metal. These are examples of components normally machined from stampings.

Courtesy Messrs. Rolls Royce, Ltd.).

Fig. 5.—Precision castings in stainless steel for general engineering purposes.

These castings are made in special stainless steel containing 14% nickel, 25% chromium and comprise miscellaneous castings used on turbo-jet engines.



Courtesy Messrs. Rolls Royce, Ltd.).

Fig. 6.—Precision castings in stainless steel (14% nickel, 25% chromium) for general engineering purposes.

Pump impeller, car door and lock mechanism components, etc.

taper cannot always be provided to ensure sound castings. In such cases, as a last resort, it may be necessary to adopt some positive method of controlling solidification.

Some months ago, it was found necessary to give some thought to this problem and a method was devised for controlling the solidification of thin metal sections in high-melting point alloys, as outlined in Fig. 1. A small H.F. melting furnace taking current at 400,000 cycles was constructed, as shown at A, with the coil extended well above the top of the crucible. The use of such high frequency current ensures very rapid melting and the absence of that stirring-action normally associated with lower frequencies. When the metal is melted, the mould to be cast is placed on top of the crucible, as shown at B, and clamped in position. Pouring is effected, with the power still on, by inverting the whole assembly, as outlined at C. When casting moulds in the usual way, there is a considerable drop in the temperature and fluidity of the molten metal on entering the mould cavity, but by this arrangement it is perfectly feasible to maintain, or if necessary increase, the temperature and fluidity of the molten metal inside the mould. Finally, progressive solidification of the cast article is assisted by allowing the mould slowly to drop clear of the heating coil as shown at D. Incidentally, one might suggest that this method should prove excellent for casting high-grade ingots in special alloys for forging and rolling purposes. Furthermore, little ingenuity would be required to adapt the arrangement for vacuum melting or air-injection casting methods.

It must be appreciated that there are size and shape limitations for all precision castings consistent with standard foundry principles so far as gating and feeding methods are concerned. To ensure production at lowest cost, castings must be designed to give the foundryman every chance of producing sound castings and the best way of doing this is for the designer to discuss the matter with the foundry at an early stage in the development of any new project. It is not usually advisable to cast threads except in very special circumstances. In certain designs some difficulty may also be experienced in reproducing accurate circular holes as there may be a tendency for ovality to occur.

So far as dimensional tolerances are concerned, a figure of plus or minus 0·005 in. may be considered satisfactory for normal working with plus or minus 0·003 in. as the best that can be offered in special circumstances. For more complete information on design factors, reference should be made to recent papers published in the technical Press.[3, 4]

(*c*) Effect of type of alloy employed.

For the economic production of precision castings, it is most essential that the alloys employed possess good castability. Considerable difficulties have arisen in this connection in the development of cast parts for the gas turbine from the special alloys developed for this service. This has been largely due to lack of knowledge as to how these new materials behaved in the foundry, and it has been necessary to gather experience in this direction. This has now been obtained and to-day founders in a position to produce precision castings in this country have a fair idea of the castability of the various alloys they may be called upon to melt. In general, high melting-point alloys containing elements liable to form tenacious oxide films, such as aluminium, are difficult to handle, particularly if the castings are subject to exacting inspection methods. There are, however, a wide range of carbon and alloy steels, cast iron, nickel and other special alloys available with good castability and eminently suitable for the economic production of any precision castings that may be required in the general engineering field.

(*d*) Ultimate cost of precision castings.

The present cost of the majority of precision castings produced is high. This can be expected in view of the large amount of development work that has had to be done in conjunction with the fact that nearly all of the castings so far required have been for specialised applications and subject to exacting inspection methods. As mentioned previously, a yield of 30% of usable castings

3 Geist and Kerr, " The Principles of Precision Investment Casting," *Foundry Trade Journal*, 1947, LXXXII, 247–254, 269–273, 291–296.

4 Sweeny, W. O., " A Realistic Appraisal of the Precision Investment Casting Process," *Iron Age*, 1947, CLX, Nov. 6, 86–90.

Fig. 7.—Alloy steel dies made by precision casting methods

(*Courtesy Messrs. Trucast, Ltd.*).

has, in many cases, been the best that could be expected. Obviously, if a higher percentage of usable castings could be obtained, say 90%, then it is possible that the cost could be reduced to one-third of what it is to-day. Here is where the founder needs the help of the engineer in developing "bread and butter" business in precision castings along more economic lines than has hitherto been possible.

In the development of this "bread and butter" business, it is suggested that the following points should be kept in mind :—

1. Select components which are not subject to too exacting inspection methods.
2. Allow the widest possible dimensional tolerances permissible.
3. Design the casting in consultation with the foundryman so that there is every chance of it being produced economically.
4. Give the foundryman full information on the properties required from the casting and the service it must withstand so that an alloy of good castability can be selected.

A precision casting is more costly to produce than a sand casting. However, in all fairness the cost of a precision casting should be compared with the machined part it is intended to replace.

The Application of Precision Castings to General Engineering

The application of precision castings in high-melting point alloys can include such articles as :—Small gear wheels ; small pump parts, impellers and rotors ; small manifold and pipe fittings ; parts of automatic mechanisms ; instrument parts ; small nozzles for burners ; gas turbine blades and nozzles ; compressor vanes ; magnets ; tools (e.g., milling cutters, reamers, twist drills, etc.) ; valve parts ; rocker arms ; cams ; sprocket wheels ; sealing rings ; metal moulds and; dies for glass and plastic industries ; knife edges ; ornamental and trade-mark motifs ; filtering equipment, etc.

The examples illustrated in Figs. 2–7 clearly demonstrate the utility of precision castings in general engineering and are worthy of close study. There is undoubtedly a place in engineering for the precision casting and future commercial development will largely depend upon close co-operation between the engineer and the founder along the lines indicated in this paper. When this is obtained, it will be found that, so far as this subject is concerned, then—"everything adds up."

The author is indebted to the Directors of The Mond Nickel Co., Ltd., and in particular to Dr. L. B. Pfeil, O.B.E., for permission to publish this paper.

Surface Finishing of Aluminium and its Alloys

THE finishing processes proper used for aluminium and its alloys are divided into four main groups—viz., mechanical processes, chemical processes, electroplating, and paint finishes. The success of each process, however, is dependent upon the efficacy of preliminary cleaning and degreasing of surfaces to be finished. It is not surprising, therefore, that, in this excellent publication, the opening section describes processes of cleaning and degreasing and emphasises the importance of thorough preparation to obtain successful results.

The mechanical processes are the well-known ones such as grinding, sand and shot blasting, scratch brushing, barrel polishing, emery finishing, and hammer finishing, used for finishing other materials, but as in other sections in this publication, modifications made necessary by the characteristics of aluminium and its alloys are clearly indicated. Attention is also directed to pattern rolling, and some excellent examples are given of designs on plates that are available for a wide variety of purposes.

The chemical processes described in one of the sections are distinct from those used solely or primarily as pre-treatment for painting, they deal with the M.B.V. process, colouring the film so produced, and matt finishing. It should be noted that a further publication describes the anodising and dyeing processes in detail, only a brief note is given in the present publication. The electroplating of aluminium and its alloys is described in considerable detail, particular attention being directed to surface preparation after which are given the plating solutions and conditions of treatment.

Under paint finishes emphasis is again laid on the importance of careful and appropriate pre-treatment, also upon the application of the appropriate priming coat following which normal painting methods can be used. The processes of lacquering, stoving, vitreous enamelling and radiant-heat drying conclude the main text of the publication. Included, however are a selected bibliography and by two lists of relevant official Specifications. Copies of the Information Bulletin No. 13 are available on application to The Aluminium Development Association, 33, Grosvenor Street, London, W. 1.

Metallurgy and Atomic Energy

By C. Hubert Plant, F.I.M.

In this, the fourth article of the series under the above heading, the author, in his consideration of the benefits to mankind likely to accrue from the use of atomic power, directs attention to its possibilities in metallurgy. Whether atomic energy will enable the metallurgist to develop new alloys or greatly improve present-day metallurgical processes only the future will tell, but these questions are discussed with the object of clarifying the outlook.

AN endeavour has been made, of necessity in a very sketchy manner, to give some idea of the present state of affairs in the realm of atomic physics. And now the question arises: Is there any possible practical advantage to be derived from all the knowledge which has been accrued in those years immediately prior to and during the second world war, apart from the obvious destructive advantage to any belligerent of a future war who is the possessor of the atomic bomb? It will have been noticed that this destructive phase of the subject has been carefully omitted; this has been done purposely because it is the use of atomic power for the benefit of mankind that is being considered rather than its destructive effects in war. But the branch of the subject with which we are now particularly concerned is metallurgy. Will the metallurgist of the future be able to wield and work wonderful new elements or alloys or will atomic energy be utilised to improve the present-day metallurgical processes?

There are three avenues of approach to the answers to these questions. We have to consider them from the standpoint of the structure of the atom when its electronic structure is included: it must be considered in the light of knowledge gained from the bombardment and breaking up of the nuclei of atoms and finally from an angle which has not yet been mentioned, the capture by the nucleus of bombarding missiles.

First, let us take the atom as a whole. Now all the properties of any element or alloy with which we are familiar are decided by the electrons surrounding the nucleus of the atom and the nucleus itself plays no part in this direction, so it is necessary to consider shortly how the electrons are distributed around their orbits. The number of electrons which can complete any one orbit, or, to use the more correct term, any one energy level is not the same for all levels. The stable grouping of electrons is in accordance with the term $2n^2$ and may therefore contain 2, 8, 18, 32 or 50 electrons in accordance with n being equal to 1, 2, 3 or 5 and the successive periods in the Periodic Table are due to this fact. The matter is, however, not quite so simple as some of the energy levels or orbits are elliptical with the nucleus located at one of the foci. This being the case, the electron will have to move more quickly at some parts of its orbit when it approaches nearer to the nucleus, which in accordance with Einstein's Theory, will increase its mass. This in turn causes the electron to over-shoot its elliptic path a little and to move further round the nucleus each time, which will give a rotating effect to the ellipse. From considerations of atomic spectra it has also been shown that the electron spins around on its own axis. Now, the original Rutherford-Bohr model of the atom did not account for these somewhat complicated motions, hence, although for many purposes the Rutherford-Bohr atom is still satisfactory, it was not until de Broglie, Heisenberg, Schroedinger and others had applied the quantum theories of Planck and Einstein to the subject under the mathematical title of "wave mechanics" that many of the properties of the atom could be explained. However, these theories are extremely abstruse and beyond the scope of these articles, but we must mention some of the results.

It has already been stated that the stable grouping of electrons is 2, 8, 18 or 32. Any group which contains either too few electrons to fill a group or any atom which contains one or two electrons over and above a full group are generally unstable. Space will not permit of a discussion of the grouping of all the elements, but as we are concerned with metallurgy the details of a few of the metals will be considered. Again, details of the main groups only are given omitting the arrangements of sub-grouping inside each main group in accordance with the quantum numbers. The following table shows the atomic number, symbol and the number of electrons in the various groups:—

Atomic No.	Symbol	Group I	Group 2	Group 3	Group 4
3	Li	2	1	—	—
12	Mg	2	8	2	—
13	Al	2	8	3	—
19	K	2	8	8	1
24	Cr	2	8	13	1
25	Mn	2	8	13	2
26	Fe	2	8	14	2
27	Co	2	8	15	2
28	Ni	2	8	16	2
29	Cu	2	8	18	1
30	Zn	2	8	18	2

An examination of this table will at once show that in every instance there are one or two electrons only in the last group. Also, it will be noticed that from potassium to nickel the third group does not fill up before the fourth group begins to fill. This is rather a complication on the part of these elements, but the reason for this need not concern us now. The point which has to be noticed is that the odd electrons in the outermost groups are very loosely held by the atom, are termed the valency electrons and actually are responsible for the element being a metal. The fact that they are loosely held means that they can easily be claimed by any other element which is not stable in its grouping, the result being that the valency electrons are shared by two or more nuclei and a chemical compound can result. In other words, it is these outer valency electrons which are responsible for the chemical properties of the element. It can, for example be shown that it is to the advantage of the atoms in order to increase their stability that two

atoms of iron should combine with three atoms of oxygen to form iron oxide. By sharing electrons in the molecule of ferric oxide, there is great stability which takes considerable force to upset. For the same reason, iron, under certain circumstances which are well-known, is very agreeable to taking a share in the management of the electrons of carbon to form iron carbide. On the other hand it must be pointed out that most of the physical properties of metals are derived from the manner in which the atoms coalesce together to form the solid crystal and the conglomeration of these crystals gives us the metal or alloy as it is usually known.

A phenomenon which has been of the greatest possible use in the study of metals for many years is that of X-rays. It is hardly necessary to remind ourselves that when a beam of cathode rays or electrons strikes a metal anti-cathode, X-radiation is emitted and the great technique of X-ray analysis has been built up following the original work of Von Laue in 1912 and that of the Braggs a little later. Here again it is the orbital electrons which are responsible for the emission of this radiation, but in this instance it is those electrons which rotate in the innermost groups which are mainly responsible, as distinct from the superficial electrons which are responsible for the optical spectra and, as we have already explained, for the chemical properties. On the other hand, particularly when secondary radiation considered, some of the outer groups do contribute.

The question which now arises is—is it possible for the physicist to so alter the arrangements of electrons surrounding a nucleus as to give to a metal or alloy properties which it does not normally possess, or to give it enhanced properties which will be useful to the user of the material?

Alternatively, is it possible to remove some electron from their orbits, and by this means provide some new form of element or alloy?

Now the metallurgist, by means of all the devices at his command can, in a very large measure, arrange the structure of his metals as he so desires, whether it be by quenching, hardening, alloying and so forth, but in no way can he or does he alter each individual atom forming the crystalline body, except in so far as the application of heat causes some exertion, first in the molecules and then, if great enough on the part of the electrons producing their "kangaroo jumps" as Jeans has described it, which emit radiation in accordance with the quantum theory already described. Let it be supposed, however, that an electron is successfully removed completely from an atom. What then has actually happened? The atomic weight will remain the same, and there will be the same positive charge in the nucleus which has not been affected. It may, therefore, seem that as the outer electrons are responsible for the chemical properties of the element, in such a case one may obtain a substance with chemical and other properties different from the original atom. If this were the case, the complications of metallurgy would certainly be increased a thousandfold, but up to the present, Nature has ordained it otherwise. Two main factors affect this possibility. First, the new atom would possess an excess positive charge, so electrically it would be unstable and would attract another electron to produce stability as quickly as possible. Secondly, there are always sufficient wandering electrons or negative charges to make this simple. These two factors work together and prevent the occurrence of such "half elements" or isobares in practice. This last statement does require some slight modification, however, as there have been ten instances known where atoms affected in this manner coalesced together to form pairs, but even these were completely unstable. We must, however, remember that light, and particularly that in the ultra-violet range of the spectrum, if allowed to fall on the alkali metals, ejects electrons from the surface of the metal. The main elements thus affected are lithium, sodium, potassium, rubidium and caesium and in every case these elements contain one only electron in the outermost group. This is, of course, the valency electron and the phenomenon is well known as the photo-electric effect, but here again, the nucleus is not affected and a new electron is quickly captured to make up the loss and to provide a repetition.

From this survey of the electronic constitution of the metallic elements it appears, therefore, that although there is a wide knowledge of the general structure of the atom in this direction, there can be little possibility of new developments in the theory and practice of metallurgy. Even the most advanced mathematical theories give no hint that there is to be any possibility of new structures, alloys or properties produced as the result of such knowledge.

And now to turn to the nuclei themselves. Mention was made earlier that in certain cases it was as if metallurgy had gone crazy. Beryllium can become carbon, boron can be turned into lithium and the latter into hydrogen gas, while as will be seen later, manganese can become iron and sodium can become magnesium with aluminium turning into phosphorus. Put like this, it appears as if nothing in the world is certain and that sooner or later the metallurgist-physicist will be able to produce at will many kinds of wonderful alloys. Undoubtedly the imagination of a Jules Verne or H. G. Wells would be able to create some very thrilling plots from the matter which could be put into their hands in this direction. But before committing ourselves to any definite opinion it will be as well to examine a few of these transformations brought about by bombardment of nuclei. Here are three transformation "equations":

$$Na^{23}_{11} + H^{1}_{1} \rightarrow Ne^{20}_{10} + He^{4}_{2}$$

$$Be^{9}_{4} + He^{4}_{2} \rightarrow C^{12}_{6} + n^{1}_{0}$$

$$B^{10}_{5} + He^{4}_{2} \rightarrow N^{13}_{7} + n^{1}_{0}$$

First sodium bombarded by a proton produces neon and an alpha particle or helium. The second case shows beryllium attacked by an alpha particle producing carbon and ejecting a neutron and thirdly boron when struck by an alpha particle results in nitrogen with the expulsion of a neutron. The first point to be noticed is that when an atom is transformed by means of a proton, the resulting element, apart from the alpha particle is lower in the atomic scale than the element attacked. On the other hand, when the alpha particle is the attacking body, the new element is one higher in the atomic scale with the ejection of a neutron. The third case shows the same thing taking place, but in this instance we have nitrogen of mass 13 and there is in nature no isotope of nitrogen of such a mass. Other instances of this kind of transformation are known and at first sight it may appear that here, at any rate, is a chance of producing some substance which is the same but different as a well-known element. This, alas, is not the case, however. In the case just cited this peculiar nitrogen is not stable and quickly transforms itself into a stable

isotope of carbon with mass 13, at the same time ejecting a positron, that is, an electron with a positive charge. This is the final reaction :

$$N_7^{13} \rightarrow C_6^{13} + p_1^0$$

And this brings us to the phenomenon first noticed and afterwards very thoroughly investigated by Fermi, the Italian physicist. He noticed that in many instances when the transformation led to the formation of a substance higher in the atomic scale, that substance was radio-active, and sooner or later spontaneously broke up into a more stable element. This phenomenon became known as artificial radio-activity. It will be noticed that in all the three instances quoted, the bombarding unit was a proton or alpha particle, but Fermi commenced to use neutrons as his bullets and obtained at once some very interesting results. Hitherto it had been found very difficult, in fact almost impossible, to disintegrate nuclei whose atomic mass was above calcium (atomic number 20), but immediately neutrons were used as the projectiles it was easily possible to disintegrate elements of the highest atomic mass right up to, as everyone knows, the heaviest of all, uranium. This artificial radio-active phenomenon has already been proved to have wide uses and has become a commercial possibility, but before discussing it in more detail a few words on the disintegration of nuclei by means other than neutrons is necessary. Although elements can be disintegrated, it should always be borne in mind that the action only produces another element in the periodic table and does not in any way produce something which has never been met before. In fact this is hardly possible as every atomic number has been filled from hydrogen at place I to uranium at 92 so whether the transformation results in a movement up or a movement down the Periodic Table, it will merely transform one element into another equally well known. Furthermore, any liberation of energy as the result of these transformations is, as has already been explained, more than counterbalanced by the greater amount of energy which has to be put in, apart altogether from the difficult procedure and costly apparatus required.

Now to turn to the so-called radio-active transformations. Here the word " so-called " has been intentionally inserted because the radio-activity is not the same as the radio-activity emanating from the true radio-active elements such as thorium, radium, etc., but the word is used to distinguish between a stable isotope of an element and one that is entirely unstable and which of itself changes to something else in a very short period of time. Consider first, the case of aluminium bombarded by an alpha particle. The first reaction is as follows :

$$Al_{13}^{27} + He_2^4 \rightarrow P_{15}^{30} + n_0^1$$

That is to say, the aluminium changes into a substance with an atomic number of 15, and we know that phosphorus is the only element with this atomic number. It must be remembered that the atomic number is the important factor as it is a measure of the number of electrons surrounding the nucleus and those electrons determine the chemical composition of the material. But there is no isotope of phosphorus with an atomic mass of 30. What happens now is that this peculiar substance which is very unstable, in order to put matters right, shoots off a positive electron or positron and rids itself of one positive charge, thus reducing the charge to 14 but with a mass still of 30, which is one of the recognised isotopes of silicon. Now to take one other example, if sodium is bombarded by the nuclei of heavy hydrogen, deuterons as they are termed, there is a transformation and a proton is ejected. Here it will be observed the atomic mass has been increased, but the atomic number is the same in accordance with this reaction :

$$Na_{11}^{23} + H_1^2 \rightarrow Na_{11}^{24} + p_1^1$$

In parenthesis it may be mentioned that it is sometimes the practice to use the symbol D for the deuteron instead of the usual hydrogen symbol H. Now there is no isotope of sodium with a mass of 24 and again there is an unstable substance which quickly ejects an electron, usually termed a beta particle, but in this instance it also gives rise to a form of radiant energy known as a gamma ray. This reaction is shown by the following equation :

$$Na_{11}^{24} \rightarrow Mg_{12}^{24} + \text{beta} + \text{gamma}$$

The fact that a negatively charged electron has left the atom increases the positive charge by one, producing an atomic number of 12 with the mass remaining constant at 24. This is one of the well-known isotopes of magnesium.

But there is a much more important method of obtaining artificial radio-activity and that is by shooting into the nuclei of atoms the particle of mass I, containing no charge—the neutron. When this is done, the neutron remains inside the nucleus and consequently increases the atomic mass by I without affecting the positive charge on it. By this means the sodium reaction, to which reference has just been made, is somewhat simplified although the results are exactly the same. Another interesting example of neutron bombardment and capture is to use manganese, the neutron producing a radio-active isotope of manganese which does not normally exist and which quickly throws off a beta particle, turning itself into the isotope of iron of mass 56.

Now it may appear that all this is very complicated and far-fetched but this is not the case. For one thing, these radioactive isotopes provide a means of obtaining radioactivity to a much more prolific extent than can be obtained by using the generally accepted radioactive elements, radium, thorium, etc. Furthermore, neutrons can be obtained in tremendous numbers and can be fired at the required elements at any velocity which may be necessary. In addition to this, these radio-isotopes are already proving themselves to be of considerable use and importance in chemistry, biology and other direction and can even now be manufactured on a commercial scale. But for our present purpose, the important question is—can they be of any advantage to the metallurgist ? The answer is very definitely in the affirmative, and although as yet, these radioactive isotopes have not been put to any practical advantage in this country, it can be confidently prophesied that, if general conditions become favourable, it may not be long before advantage can be taken of them. It is unnecessary to remind metallurgists of the importance of the diffusion of metals or of the diffusion of gases in metals. In the production of steel ingots, in the manufacture of both ferrous and non-ferrous alloys, in case-hardening or in welding, to name a few processes, it is of great importance to know how and to what extent one element will diffuse into another or alternatively how one phase of any element will diffuse into another of its own phases. If, therefore, a radioactive isotope is prepared of the element which it is desired to examine, we know from what has already been said that this will

be chemically exact with the stable form of that element. By introducing a very small amount of the isotope with the bulk of the element, this will not affect the metal or alloy in any way but it will disclose its presence in the metal and can easily be detected by one of the instruments now commonly used for this purpose. Information can then be ascertained as to how that element is migrating or diffusing through the metal as a whole, its rate of diffusion and dispersal through the bulk which will be of undoubted benefit. Very small percentages of inclusions are often a source of great trouble to the metallurgist. Here again, the addition of extremely small amounts of the suspected inclusion in a radioactive form to the melt of metal can then be detected by microradiographic technique and this will point out the position of the suspected inclusion. Yet again, it is very often that extremely small percentages of elements occur in ferrous and non-ferrous alloys. They are extremely difficult to identify either by chemical analysis or by micro-examination. A radioactive form of the suspected element will disclose its presence and therefore the presence of the suspected impurity. At the moment, it is too early to state that even the percentage of the impurity itself can be calculated, but it appears highly probable that ultimately a technique will be discovered whereby even this may be done.

Reference has lately been made to neutron capture and its results, which naturally introduces that branch of the subject which has been in so many minds during the last two years and to a much greater extent in a comparatively few minds for the past eight years. We refer to atomic energy as applied to uranium and its kindred elements, but not from the standpoint of destruction and war.

Uranium was first isolated by Perigot in 1842. It can be obtained from pitchblende and uraninite, both oxides of uranium, also from carnotite, a vanadate of potassium and uranium, from polycrase containing yttrium and uranium and from other minerals. As may be expected from the heaviest of the elements, it is radioactive and at one time was thought to be the ancestor of radium, but since the preparation of plutonium it seems fairly certain that this is an older ancestor of the " uranium family " which has, during the millions of years of atomic evolution completely decayed away leaving uranium as the heaviest element. In fact, there may have been still heavier elements from which plutonium descended. Uranium did not really come very prominently into the news until the discovery of radioactivity by Becquerel, but it was not a first line attraction until Otto Hahn discovered the phenomenon of fission in 1938, which was confirmed later by Lise Meitner. Hahn had subjected uranium to bombardment by neutrons and obtained a very surprising reaction, the resulting substance being chemically identical with the element barium of atomic number 56. Now barium occurs not far from the midway line of the Periodic Table and Lise Meitner at once suggested that the uranium atom had been split into two. This was confirmed by experiment, and it was then discovered that the energy liberated by this split amounted to something of the order of 200,000,000 electron volts. Further experimenting led to the fact that the elements immediately below uranium were also subject to fission but that those elements a little lower in the scale of atomic numbers were not so affected.

It will be remembered that when discussing atomic energy it was explained that the elements at the top of the list were liable to this phenomenon because the disruptive repelling force of the positive charges would be greater than the cohesive force of surface tension, consequently it only required the shock of the bombarding neutron possessing comparatively little kinetic energy to bring about the splitting of the atoms, and the release of an immense amount of metastable pent up energy. Another important point connected with this process of fission is that only neutrons possessing considerable velocity will effect the fission. The balance of forces within the uranium atom is so fine that if neutrons of slight velocity are used, they have not the energy to cause the upset, but on the other hand if they have too much energy they may not be captured by the uranium atoms, lose some of their energy and so a great amount of energy is wasted to no purpose.

So far we have referred only to the common isotope of uranium with an atomic mass of 238, but there is an isotope of mass 235 which only forms 0·7% of natural uranium which acts very readily to thermal neutrons. " Thermal neutrons " by the way, are neutrons which are moving at speeds of the molecules in water at ordinary temperatures. The reason for this is that 1,000,000 electron volts less are needed to produce fission in uranium 235 than in the 238 isotope. Obviously, it is therefore better to use the former, but the difficulty is that there is so little of it and further, it is very difficult to obtain.

Investigation into the reactions connected with uranium 238 led, however, to some very important results. It was found that when a neutron is shot at this isotope and is captured by it, the change is to a very unstable isotope of uranium with a mass of 239 while a photon of radiation is emitted. Uranium 239 quickly shoots off an electron, thus increasing the positive charge and atomic number to 93 producing a substance to which the name Neptunium has been given. This in turn is also unstable and discharges both an electron and a photon of radiation or gamma ray. The net result is still another substance with mass 239 and atomic number 94, named Plutonium. The great importance of this is that it is comparatively stable and undergoes fission by slow-moving electrons, the energy emitted being in the region of 200,000,000 electron volts. Not only this, but it is also subject to the chain reaction phenomenon which has already been explained. It will be interesting to note that during the complete fission process, there are no less than eight different adjustments of artificial radioactive substances before the final stable nuclei are reached. During those processes there is in every instance the emission of some charged particles or radiation in the form of photons, and it is one of the marvels of these reactions that they all take place in an infinitesimal fraction of a second. Plutonium, to produce an Irish bull, is heavier than the heaviest known element, but quite recently one of the discoverers of plutonium, Dr. G. T. Seaborg has succeeded in bombarding this metal with helium nuclei and has obtained two other substances having atomic numbers of 95 and 96. They are naturally unstable and little has been learned about them, but in the meantime they have been christened Americium and Curium. As has already been stated, it appears that plutonium must have occurred in the dim past history of atoms and now we have two other still more remote ancestors produced by human scientists.

Are we now any nearer to obtaining an answer to the question—what is Energy? Everything certainly seems to point to the suggestion that free Energy is the basis of all matter in the universe and that it is akin to what we term light or more exactly to radiation. Under certain circumstances there appears to be something approaching a condensation of this energy producing what we recognise as electrical charges. Why some should be positive and some negative is still a mystery but, as has been seen, combinations of the two types produce atoms which in turn produce the matter of the universe as it is known and experienced by us human beings. Perhaps it may be given to the physicist-metallurgist of the future to realise how energy can be so condensed to produce matter and when that day arrives, the last secret of creation will have been mastered.

There is one last thought. Some attention has already been paid to the energy available by the fusion of nuclei of elements in the lowest places in the Periodic Table. If helium is produced from hydrogen nuclei, the energy emitted when calculated in ordinary units would be something of the order of a quarter of a million horse power per hour per gram of helium. It is now believed that this is the manner in which the sun maintains its heat. From the practical standpoint, the matter is hardly yet outside the imaginative state, but it appears that there we again have a fruitful field for research with the ultimate possibility of the source of an endless amount of energy probably derived in a more simple manner than from the fission of uranium or plutonium.

That human beings are now in possession of many of the secrets of the working of the universe is without doubt. There is still much more to learn and to understand, some of which will undoubtedly be achieved, but the dangers are so immense that unless the whole subject is treated from the viewpoint of increasing benefits to mankind, there is not the slightest doubt that one day human civilisation may be entirely annihilated.

Staff Changes and Appointments

MR. W. R. BERRY, M.Sc., M.I.Mech.E., F.R.I.C., F.I.M., recently resigned from the Board of Messrs. Ibbotson Brothers Ltd., Globe Steel Works, Sheffield, and from his appointment as Chief Development Engineer to the Whitehead group of companies. He has opened a practice as Consulting Engineer and Metallurgist at Grey Garth, Wynmore Avenue, Bramhope, near Leeds.

MR. H. BOOTMAN has left the Department of Research and Technical Development, Messrs. Stewarts and Lloyds, Ltd., Bilston, Staffs., to take up an appointment as Metallurgist in charge of melting and refining with H. J. Enthoven and Sons Ltd., Darley Dale Works, South Darley, Matlock, Derbyshire.

MR. JOHN O. EDWARDS, M.Sc., A.I.M., has relinquished his position with The British Aluminium Company Ltd., to take up an appointment as metallurgist with the Mineral Dressing and Metallurgy Division of the Bureau of Mines, 568, Booth Street, Ottawa, Ont., Canada.

THE RIGHT HON. THE VISCOUNT FALMOUTH has been elected Chairman of the Imperial College of Science and Technology, in succession to the late Lord Rayleigh.

MR. JOHN FRY, who founded the business in 1912, is retiring from the position of Chairman and Managing Director of Fry's Metal Foundries Ltd.

MR. N. J. GRIFFITHS has been appointed Works Manager at the Brownhills Works of Cindal Aluminium Ltd., Lichfield Road, Brownhills, Staffs.

MR. H. O. LUDKEY has left B.K.L. Alloys Ltd., Kings Norton, Birmingham, to take up the post of Manager of the Aluminium Melting Works, Aerlec (Aluminium) Ltd., Stoke Prior, Bromsgrove, Staffs.

CAPT. I. MINKOFF, B.Sc., who was released from H.M. Forces in August last, is now employed in the Metallurgical Laboratory of the De Havilland Aircraft Company.

MR. WILLIAM B. BAXTER, general manager of Appleby–Frodingham Steel Company, has been appointed a director of United Steel Companies Ltd. He joined the Frodingham Iron and Steel Company, now the Appleby–Frodingham Steel Company, and after assisting the late Mr. James Henderson became works manager in 1927 and general manager in 1939. As from January 1st 1948, he has acted as joint general manager.

MR. D. R. WOOD, B.A., L.I.M., has left the Northern Aluminium Company Ltd., Rogerstone, Newport, to join the Research Laboratories, The Mond Nickel Company Ltd., Wiggin Street, Birmingham.

MR. A. F. DIXON has retired from his position as chief engineer of the Brightside Foundry and Engineering Co. Ltd., Sheffield, but will continue to serve the company as a consultant.

MR. B. M. HARDMAN has recently been released from H.M. Forces and has been appointed lecturer in Metallurgy at the North Staffordshire Technical College, Stoke-on-Trent.

MR. E. J. D. SMITH has left the Motor Industry Research Association and has taken up an appointment as scientific officer in the Refractories Section of the British Iron and Steel Research Association.

MR. E. W. HANCOCK, M.B.E., is joining the Board of Humber Ltd., Coventry. He will be undertaking the duties of general manager (Works) commencing April 1st, 1948. Mr. Hancock has been works general manager with Rubery Owen and Co. Limited, Darlaston, since August, 1942, having previously held the appointment of Works Manager, Humber Limited, Coventry for six years.

MR. C. G. HOPPER has resigned his position as advertising manager of the Boulton and Paul group of Companies and his plans at the moment are fluid. He was formerly advertising manager, for seven years, of the Simmonds group (Simmonds Aerocessories Ltd., Simmonds Products Ltd., etc.).

MR. F. W. ROWE, B.Sc., has been elected Vice-Chairman of the British Steel Founders' Association.

DR. E. W. FELL, Dr. Ing., M.Sc., F.R.I.C., F.I.M., has been appointed senior assistant in metallurgy at the Bradford Technical College.

MR. E. B. HILL of Charles Hill and Co. Ltd. was elected as Chairman of the Zinc Alloy Die Casters Association at a recent meeting of the Council. Mr. Hill will be assisted by the retiring Chairman, Mr. A. E. Mills of the British Die Casting and Engineering Co. Ltd., as well as by Mr. J. W. Cartlidge of Dyson and Co., Enfield (1919) Ltd., who was at the same time elected Chairman Designate to succeed Mr. Hill at the end of his term of office. These two gentlemen will act as Deputy Chairmen.

The Influence of Chemical Composition and Quality of Raw Materials on the Physical Properties of Silumin Gamma*

By Vlad. Zednik, Ing. Dr.

The development of aluminium-silicon alloys is interesting in two ways : first, the tendency to furnish cheaper raw materials for their production, and second, the simultaneous improvement of the physical properties of the finished castings. The reciprocal influence of both these efforts and the analysis of results and experiences gained in the foundry of Skoda-works are the content of this paper.

The Swiss and Canadian raw materials used at this time were produced by alloying Al, sometimes of inferior quality, with Si or with a temper alloy containing a high percentage of Si, according to the German method. The raw German silumin on which the whole of Central Europe depended during the occupation was defective in the same way, and it was delivered ready alloyed with Mn and not with Co, because of the shortage of the latter.

Cobalt and Manganese as Compensators for Iron Content

In order to investigate the effect of substituting Mn for Co, a series of experimental silumin gamma melts was made from French alpax, each series with increasing Mg content. Si was kept at about 9%, Mn varied between 0·4–0·5%, Fe between 0·35–0·40%. Modification was carried out by the usual Skoda process, lettered "A" in the diagram, Fig. 16, and by other two adopted methods of refining, lettered "B" and "C." The results, showing the relation between the Mg content, tensile strength and the elongation, are given in Fig. 16. They show the elongation of alloys containing Mn to possess lower values in comparison with alloys containing Co. Microstructural examination showed two effects : the expected primary formation of coarse complex crystals occurring with lower Fe content, and a greater tendency to segregation. This was found by a comparative experiment on test pieces cut from an identical place in the wall of the casting, about 45 mm. thick. The results are shown in Fig. 17.

On the basis of this preliminary experiment a scheme was worked out to determine the most advantageous chemical composition of Mn-silumin gamma. This concerned mainly the Mg content, which had to be increased slightly, and the Mn content, which had to be lower than that of iron. A further series of experiments was then carried out, using the usual German raw material and a special raw material of the composition of French alpax. From the German and French experimental raw materials, 272 melts were made and tested. The results of the tests on separately cast test pieces are given in relation to the Mg content in Table I and Fig. 18. This figure contains some additional results obtained with silumin containing Co and made from German raw material, until supplies of Co became exhausted, as well as production results after the introduction of mass production of Mn-silumin from an alloyed German raw material marked "silumin beta manganese." A better illustration of the uniformity of quality is given by Fig. 19, in which the results of the tests on the mass-production melts have been plotted as integrated frequency curves. In them

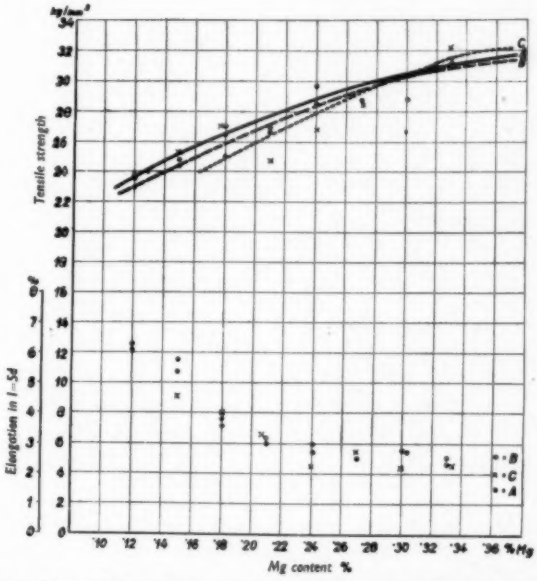


Fig. 16.—Mechanical properties of separately cast test bars, made from experimental melts of silumin gamma alloyed with manganese, in relation to Mg-content. Melted from French raw materials.

the most interesting cases are compared—namely, silumin gamma, containing Co and made from French and German raw material, and silumin gamma containing Mn and made from the French and German raw material.

Tables II and III give the results of tensile tests on test pieces illustrated in Fig. 21, prepared from the cylinder blocks and cast from the French and German raw materials, respectively. It will be seen that the results are more uniform in the case of silumin gamma containing Co and made from French raw material.

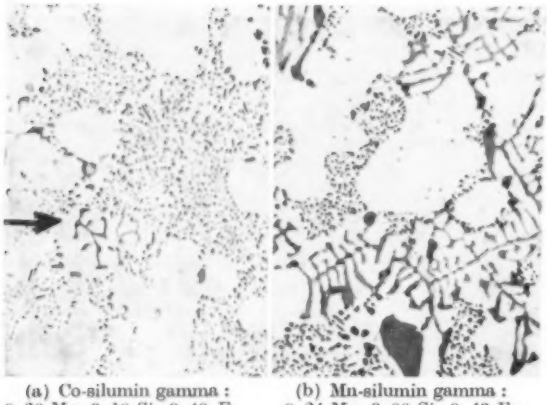
The fatigue limit of silumin gamma in bending, which was taken on a series of chill- and sand-cast test pieces from these experimental melts, showed a certain scatter of results which is evident from the following data :—

Type of silumin	Raw material	Mould	Fatigue limit in bending for 20,000,000 of cycles — kg/mm²
Co	French	Chill-cast	8·5–10·5
	French	Sand-cast	5·7– 8·2
Mn	German	Chill-cast	6·5– 8·5
	German	Sand-cast	5·0– 5·8

*Continued from page 200, February issue.

TABLE I—AVERAGE OF TESTS ON 272 MELTS

Type of silumin	Raw material	Average magnesium content	Average mechanical properties: Separately casted test bars — Yield point	Tensile strength	Elongation 1 = 5d	Cast on test bars — Yield point	Tensile strength	Elongation 1 = 5d
		%	kg/mm²	kg/mm²	%	kg/mm²	kg/mm²	%
Co	French	0·203	22·3	27·3	7·45	20·3	25·4	4·15
	German	0·219	19·3	25·0	5·43	17·7	23·0	3·03
Mn	French	0·227	23·2	27·4	4·19	21·7	25·4	2·94
	German	0·238	20·5	26·9	3·49	18·8	24·8	2·31



(a) Co-silumin gamma: 0·20 Mg, 9·10 Si, 0·40 Fe, 0·44 Co, 0·01 Mn, 0·0055 Na. x 150.

(b) Mn-silumin gamma: 0·24 Mg, 9·00 Si, 0·43 Fe, 0 Co, 0·41 Mn, 0·012 Na. x 150.

Wall thickness about 45 mm. in both cases.

Fig. 17.—The formation of complex crystals by compensation of the iron content in silumin gamma by alloying with cobalt or manganese, respectively. Test pieces cut from an identical place in the casting.

There is a smaller difference between the values of the fatigue limit of silumin containing Co and Mn respectively than between the tensile values. This furnished further support of evidence gained with other material—namely, that the dynamical properties are often not directly related to the statical ones.

Though the results of tests on experimental melts and on mass production show clearly the inferior quality of silumin gamma made from German raw material, and the deterioration caused by the substitution of Mn for Co, they do not show the increased nonuniformity, and the decrease in reliability of the mass production based on the German synthetic raw material alloyed with Mn[9]. The cause of this varying quality of the product lay in the fluctuating quality of the German raw material, the structure differed from one melt to another and it was not unusual to find differences even with the same melt. It depended on the degree of superheat before

TABLE III.—RESULTS MECHANICAL TESTS ON CYLINDER

Part of the casting tested	I. Y.P. kg/mm²	I. U.T.S. kg/mm²	I. E. 1 = 5d %	II. Y.P. kg/mm²	II. U.T.S. kg/mm²
Cylinder wall	19·6	26·8	3·5	20·8	27·3
Cylinder bottom	21·6	28·0	4·4	22·3	27·9
Water jacket	19·3	23·5	2·1	19·6	20·5
Flange	21·8	27·7	3·8	16·3	21·6
Cast-on test bars	—	—	—	20·1	24·5
Total average value	—	—	—	—	—

alloying with Si, the impurity content, the casting temperature, the temperature of the mould, and the residual Na content. As will be noted in Fig. 22, the crystals of Si were sometimes equiaxed, and sometimes acicular, with a varying degree of dispersion, dependent on the refining effect of Na. The tendency to separate in this fashion is a specific property of the material, even after remelting and modifying, and remains so until its cause is removed. This can be seen in Fig. 23 showing the microstructure of a surface layer and the middle portion of the wall of the casting. Raw materials of this character need to be modified more thoroughly and a disproportionately high Na content is necessary. This, however, increases the tendency to gasiness, irregular shrinkage, and the formation of oxide skins. Gasiness manifests itself in the form of microporosity especially in those parts of the casting which solidify slowly, and in places distant from the pouring ingate. This is the reason for microporosity in relatively quickly cooled cast on samples. (See Fig. 24). The faster decay of the refining effect, after modification by sodium, accentuates the unfavourable influence of imperfect structural homogenity. Fluctuating fineness of the Al-Si eutectic, with identical Na contents, and the differing stability of the refined condition, has naturally a very damaging influence on the uniformity of quality in the mass production of complicated and large castings.

Excessive and insufficient Mn contents, for a given amount of Fe and Ti, are more detrimental than those of Co[10]. The undesired formation of coarse complex Fe-Mn-Al-Si crystals has been noticed even with low Fe and theoretically satisfactory Mn contents as can be

9 The statistical mathematical work on this problem was completely destroyed during an air-raid.

10 The Fe-content of the tested silumins gamma fluctuated between 0·3–0·65%.

TABLE II.—RESULTS OF MECHANICAL TESTS ON CYLINDER BLOCK (DAIMLER-BENZ 605) MADE OF SILUMIN GAMMA ALLOYED WITH

Part of the casting tested	I. Y.P. kg/mm²	I. U.T.S. kg/mm²	I. E. 1 = 5d %	II. Y.P. kg/mm²	II. U.T.S. kg/mm²	II. E. 1 = 5d %	III. Y.P. kg/mm²	III. U.T.S. kg/mm²	III. E. 1 = 5d %	IV. Y.P. kg/mm²	IV. U.T.S. kg/mm²	IV. E. 1 = 5d %	V. Y.P. kg/mm²	V. U.T.S. kg/mm²
Cylinder wall	21·3	25·3	5·5	22·3	25·8	6·2	22·6	26·0	6·3	22·1	26·0	5·7	21·8	25·2
Cylinder bottom	22·3	27·0	7·4	22·3	26·9	7·6	22·5	27·1	8·6	22·3	27·4	10·0	21·6	27·1
Water jacket	21·5	24·9	4·5	22·0	24·0	3·1	22·9	24·9	3·2	22·7	24·5	3·3	22·1	25·5
Flange	22·1	26·1	6·1	23·0	25·3	6·0	21·9	25·6	5·3	22·4	26·4	5·5	21·2	26·0
Cast-on test bars	21·3	22·9	1·75	22·2	24·3	2·75	23·2	26·2	5·75	22·2	25·1	4·5	21·5	27·5
Total average value	—	—	—	—	—	—	—	—	—	—	—	—	—	—

seen in Figs. 25 and 26 taken on 11 mm. thick cast-on samples. The embrittleing effect of such a structural component is naturally detrimental and undesirable. The crystals in Fig. 26 have separated from a melt of composition 0·21% Mg, 9·48% Si, 0·31% Fe, 0·44% Mn; the ultimate tensile strength of the cast-iron test piece from which the micrograph has been taken, after heat-treatment, reached 21·9 kg./mm.² with elongation of 0·75% at l = 5d. The yield point was missing, fracture was premature and contained shining planes revealing the presence of coarse complex iron-rich crystals. The explanation of this phenomena has already been referred to. Due to the selective solidification metal running through becomes enriched in alloying elements and impurities. Microradiographic measurements taken reveal segregation of Mn either in the complex Fe-Mn-Al-Si crystals or perhaps in phases containing Al + Mn in varying proportions. The various colouring of the complex crystals obtained after etching with warm 10% H_2SO_4 or ½–1% HF can be explained by their varying composition. The shape of the complex crystals does not alter substantially on heat-treatment as their solubility in the alpha phase at normal temperatures is very slight indeed.

In the silumins alloyed with Co and made from

BLOCK (DAIMLER-BENZ 605) MADE OF SILUMIN GAMMA ALLOYED WITH MANGANESE AND MELTED FROM GERMAN RAW MATERIALS

IIII.				IV.			V.			VI.				Average		
E. l = 5d %	Y.P. kg/mm²	U.T.S. kg/mm²	E. l = 5d %	Y.P. kg/mm²	U.T.S. kg/mm²	E. l = 5d %	Y.P. kg/mm²	U.T.S. kg/mm²	E. l = 5d %	Y.P. kg/mm²	U.T.S. kg/mm²	E. l = 5d %	No. of tests	Y.P. kg/mm²	U.T.S. kg/mm²	E. l = 5d %
4·0	20·0	28·2	4·4	20·7	27·3	4·0	21·0	27·6	4·3	20·8	27·5	3·7	48	20·2	27·4	4·0
3·6	23·3	28·4	4·6	22·7	28·3	4·4	23·7	28·7	4·2	23·6	28·3	4·2	6	22·8	27·9	4·2
1·4	17·5	20·4	1·6	—	21·4	1·7	—	21·4	1·8	19·0	21·6	2·6	42	18·3	21·4	1·8
5·5	21·4	29·2	4·4	—	—	—	20·7	26·5	2·8	19·8	26·8	3·5	7	20·3	27·4	3·8
3·8	—	—	—	—	—	—	20·3	23·8	2·6	—	—	—	4	20·2	24·1	3·2
—	—	—	—	—	—	—	—	—	—	—	—	—	107	20·3	25·9	3·4

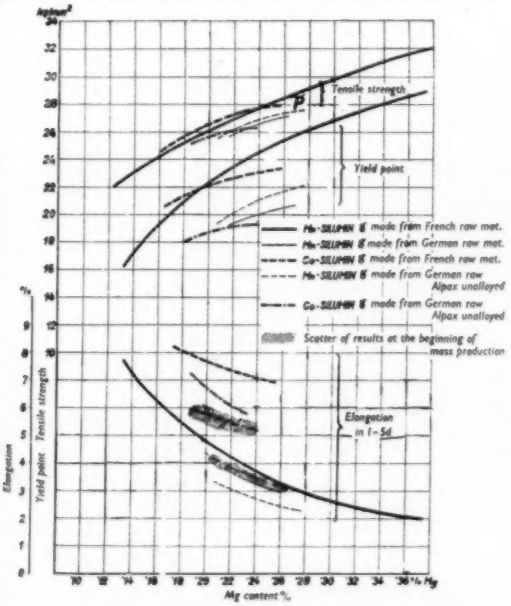


Fig. 18.—The relation of mechanical properties of separately cast bars, from experimental melts of silumin gamma, melted from diverse raw materials and alloyed with manganese or cobalt, to magnesium content and the type of raw material used.

COBALT AND MELTED FROM FRENCH RAW MATERIALS

VI.				Average		
E. l = 5d %	Y.P. kg/mm²	U.T.S. kg/mm²	E. l = 5d %	No. of tests	Y.P. kg/mm²	U.T.S. kg/mm²	E. l = 5d %
6·1	21·5	24·2	3·7	48	21·9	25·4	5·6
8·8	21·6	25·7	7·0	6	22·1	26·8	8·2
3·8	21·4	23·9	3·7	42	22·1	24·6	3·9
5·5	21·0	25·3	6·6	18	21·9	25·8	5·9
3·75	20·4	25·0	4·5	6	21·8	25·2	3·8
—	—	—	—	120	21·9	25·5	5·5

Fig. 19.—Mechanical properties of cast-on and separately cast bars of 272 experimental melts expressed by integrated frequency curves. Comparison of results on bars made from French and German raw material.

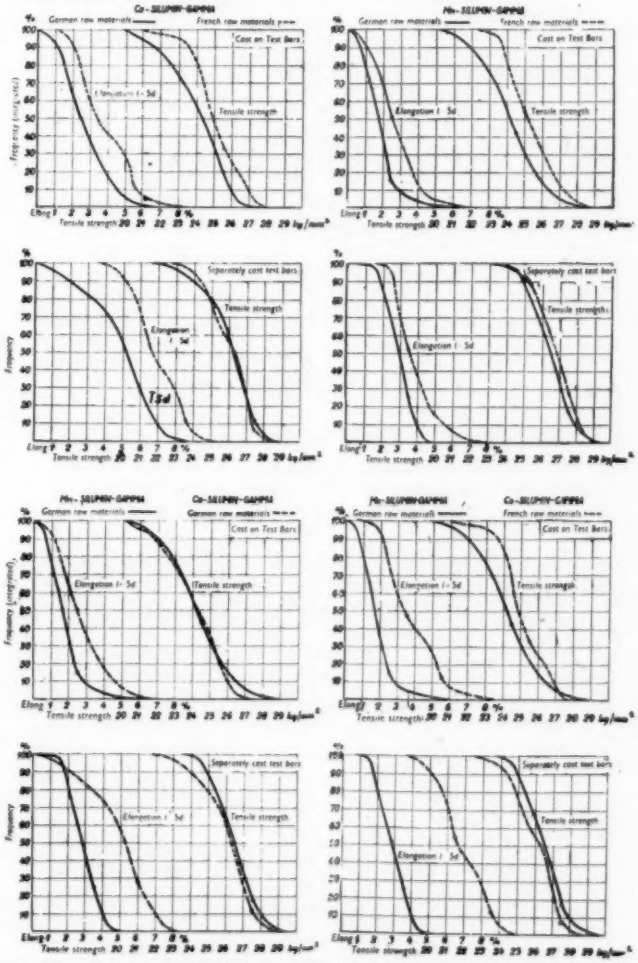
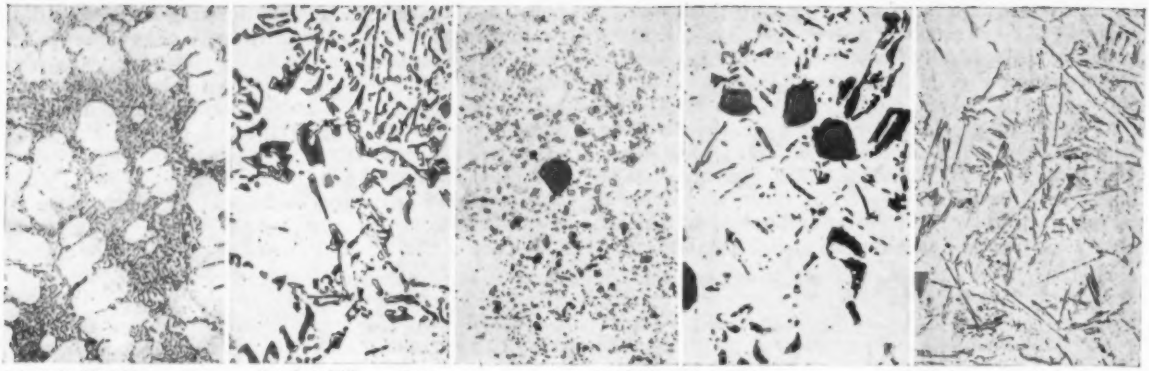


Fig. 20.—Comparison of the most interesting cases given in Fig. 19.



(a) 10% NaOH. x 100. (b) 1% HF. x 300. (a) (b) Silicium in partly " modified " form. 10% NaOH. x 100 (c) (d) Silicium in grain-like form. (e) Silicium mainly in lamellar form. 10% NaOH. x 100.

Fig. 22.—Various types of the German raw material " Silumin beta-manganese " with composition about 0·30–0·45% Mg, 9% Si, 0·40–0·50% Mn, 0·40–0·50% Fe.

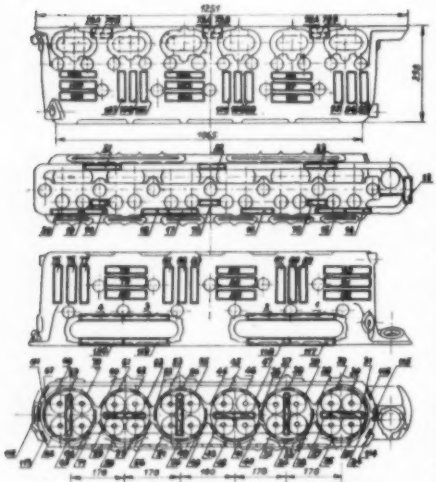
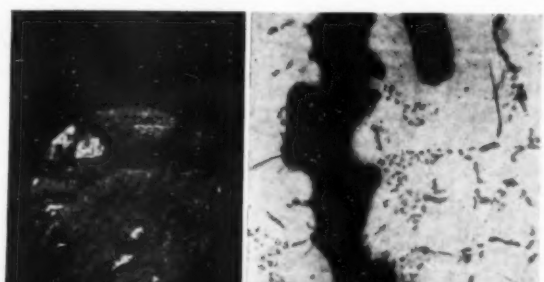


Fig. 21.—Schematical layout of the test pieces taken from the cylinder blocks of Daimler Benz aircraft engine.

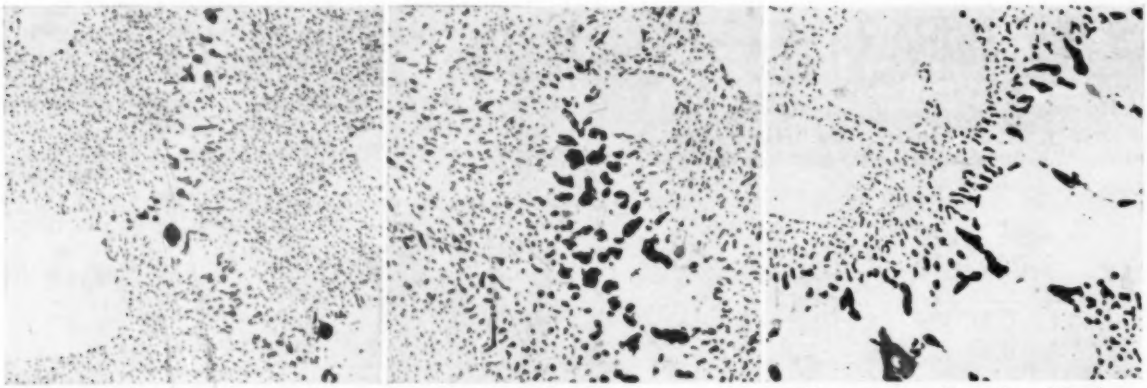
French electrolytic raw materials, the complex crystals are mostly separated in an extremely fine-grained form, and are usually embedded in the Al-Si eutectic. As the complex crystals, whether etched or unetched, differ in this form only very slightly from the Si grains it is often difficult, especially in reproduction, to distinguish them from each other, as for instance in Fig. 27. The actual shape, position and size of the crystals can be seen in Fig. 25a. It is only in the thicker wall sections that sharply edged and bigger crystals separate, and these seldom take on a skeleton-like form.

The difficulties in production mentioned above became accentuated as the war progressed, firstly due to the ever-increasing Fe content, and eventually even



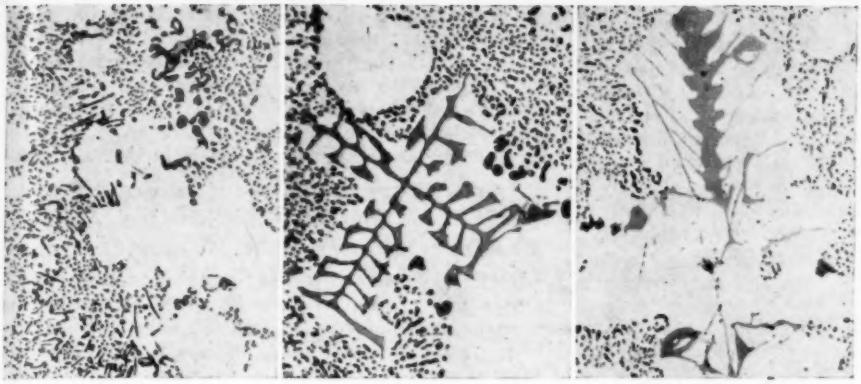
(a) White-spotted fracture after tensile test. x 3. (b) Interdendritic cavities. x 150.

Fig. 24.—Microporosity in gassed Mn-silumin gamma.



(a) (b) The tendency towards a quasiprimary separation of Si in consequence of dendritic segregation. x 150. (c) The reducing effect of modification, after some period of freezing, in the middle of a green sand casting. x 150.

Fig. 23.—Mn-silumin gamma. Coarsening of Si at the edges of dendrites.



(a) 0·23 Mg, 9·32 Si, 0·45 Fe, 0·36 Mn, 0·13 Co, 0·08 Ti, 0·0062 Na. x 150.

(b) 0·23 Mg, 9·24 Si, 0·38 Fe, 0·39 Mn, 0 Co, 0·09 Ti, 0·015 Na. x 150.

(c) 0·27M g, 9·10 Si, 0·45 Fe, 0·46 Mn, 0 Co, 0·09 Ti, 0·015 Na. x 150.

Specimens from the 11 mm. thick cast-on bars.

Fig. 25.—Various forms of separation of complex ironrich crystals in silumin gamma.

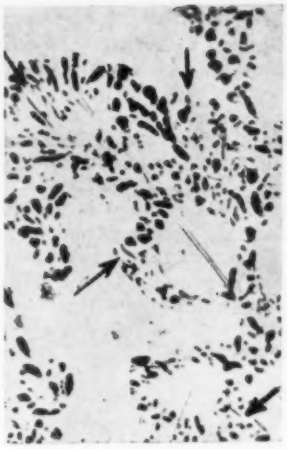


Fig. 27.—Microstructure of silumin gamma made from French raw material alloyed with cobalt. Distribution of complex Fe-Co-Al-Si crystals at 0·38%Fe and 0·44%Co content. Unetched. x500.

to the scarcity of Mn to compensate for the iron content. It was interesting to note that such silumins were rather susceptible to undercooling, resulting in the formation of alpha dendrites, non-uniform crystallisation in different parts of the casting, and a macroscopic formation of the Fe and Mn rich crystals. It is likely that the varying susceptibility towards undercooling had an influence on the refining action of modification by Na and caused irregular dispersion or patchiness of the Al-Si eutectic. Also, it either magnified or masked the influence of the original structure of the raw material and its impurities, especially Ca, P and carbides. The content of these impurities could not be determined quantitatively, however, due to the lack of the necessary apparatus, so that neither their content nor their influence could be examined accurately.

The deteriorating quality of silumin gamma containing Mn during the war can be expressed approximately by the mean values of the properties of the test pieces cast-on to an identical casting, as given in Table IV.

TABLE IV.—AVERAGE VALUES OF THE PROPERTIES OF THE TEST PIECES CAST ON TO CRANK-SHAFT CASTING FOR A "DAIMLER BENZ" ENGINE

Production year	1941	1942	1943	1944
Number of tests	785	3,760	5,616	2,845
Average yield point kg/mm²	24·2	23·1	22·4	21·3
Average tensile strength	28·6	27·4	27·7	26·5
Average elongation 1 = 5d.	4·8	4·7	4·2	3·7

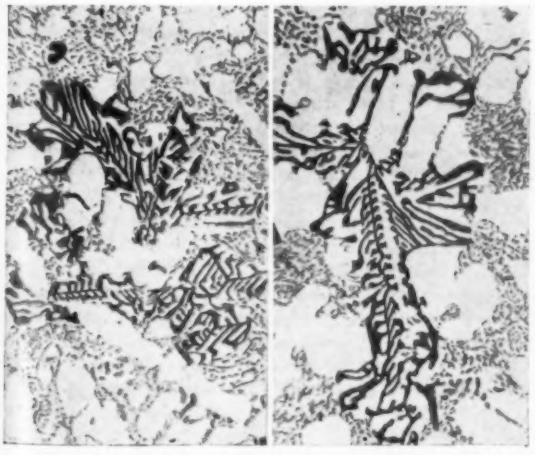


Fig. 26.—Complex iron-rich crystal formation in a 11 mm. thick cast-on bar of silumin gamma, melted from German raw material alloyed with manganese. HF x 60.

It is perhaps hardly necessary to say that the above results were obtained on sound castings only. The castings, even if defective due to the bad quality of raw material, were scrapped after radiographic examination before heat-treatment, and therefore not tested mechanically.

Summary Conclusions

The results of tests and statistics prove that the quality of silumin gamma depends greatly on the purity and structure of the raw silumin used for producing it. Raw silumin made electrolytically is usually very pure, showing highly dispersed eutectic silicon of uniform distribution, a very uniform ability to modification by sodium and a considerable stability in its modified state. On the other hand, raw silumin made synthetically, invariably shows greater deviation in the content of impurities introduced by the temper alloy, which is usually made by reduction with carbon. It often contains silicon in granular form, which is masked to some extent by treating the raw silumin with sodium. The improvement of such a product in the foundry, by the usual modification with sodium, is more difficult and the results are not uniform.

Cobalt compensates for the unfavourable influence of iron in silumin gamma better than does manganese, which has a greater tendency to segregation, and to primary formation of coarse complex crystals rich in iron and manganese. These affect the tensile strength and ductility of the alloy adversely, which has been shown by metallographic tests as well as by statistical data based on the results of mechanical tests on series of aircraft castings.

The author would like to thank the Technical Chief Manager of the Skoda Works, Plzen, for permission to publish this paper.

The Institute of Metals (*Continued from page 242*)

where l_t = length of specimen after time t; l_o = a constant which in the present tests can be taken as the original length of the specimen; β = a constant associated with the so-called "grain-rotation" type of flow. This type of flow is often called transient flow or β-flow and appears to be dependent on a rotary motion, occurring within the crystal grains or, more accurately grain fragments. It is considered to result essentially from the process of glide-plane slip; and k = a constant, representing a type of flow considered to differ (perhaps fundamentally) from β-flow. In applying the Andrade equation to pipe tests, it may be written in the form:—

$$\epsilon = (1 + \beta^{1/3})e^{kt}$$

where E = diametric extension per unit diameter = peripheral extension per unit periphery = $\frac{l_t - l_o}{l_o}$

For small values of kt the approximation $e^{kt} = 1 + kt$ leads to the approximate relationship: $\epsilon = \beta^{1/3} + kt$ from which the calculation is simple.

In the present paper, only the work on pure lead and alloy "E" (antimony 0·2, tin 0·4%, lead remainder, to B.S. Specification No. 801) is described and the application of the results to the particular problem of sheath for pressure cable is dealt with in detail. The more important conclusions reached are:—

The results of the constant-hoop-stress tests indicate that, within a similar range of creep rates, the fundamental mechanism of creep flow in the pure-lead and alloy "E" pipes under test is not greatly different. In particular, the corresponding Andrade β and k constants are of a similar order and decrease in a similar way with decreased applied stress. The possibility of certain secondary effects in alloy "E" cannot be excluded, but there is insufficient evidence to show their precise nature or importance.

For both materials, the indications are that, as the applied stress diminishes, the k constant decreases to a very small amount, while the β constant still has an appreciable value, i.e., the proportion of β-flow markedly increases.

The decrease in extension which the alloy "E" pipe is able to withstand before failure, and the increased liability to inter-crystalline fracture, with decreased mean creep rate may be connected with the effects of β-flow when the amount of k-flow combined with (or following) it decreases. Pure lead, it is postulated, is better able to withstand these conditions than alloy "E".

Failures in pressure-cable sheath of certain alloys may be connected with these effects and possibly also with effects, of a fundamentally similar nature, due to stress relaxation. These effects are, furthermore, aggravated by the presence of extrusion defects.

The influence of grain-size needs further study, and grain refinement may prove to be of great importance, if carried out in a suitable manner, in increasing resistance to failure by β-flow, or whatever grain-boundary disrupting influence is concerned.

The author emphasises that the theories developed in this paper are not claimed as complete. Further data will undoubtedly be necessary before a full explanation can be offered of the behaviour of alloy "E" and similar materials under prolonged creep. A crucial point, for instance, is that the effect of (rapid) β-flow, when combined with the (less-rapid) k-flow, corresponding to tests at the higher stresses, is less likely to cause failure (in alloy "E") at small extensions than the effect of slow β-flow, taking place over a long period with diminishing rapidity and combined with slow k-flow. While the evidence is in favour of this view, there is no direct proof. It is worth noting that the possible change in nature of k-flow at slow creep rates may be of importance in this respect, while the precise manner of relative grain movements at boundaries needs further study.

RECENT DEVELOPMENTS IN CORROSION-RESISTANT ALUMINIUM-MAGNESIUM ALLOYS

By P. Brenner, Dr. Ing., and W. Roth, Dr. Ing.

ALUMINIUM alloys of the aluminium-magnesium type are of special interest, since in both the supernaturated-solid-solution and the aged states they show no marked age-hardening, although the solubility of magnesium is very large and decreases rapidly with decreasing temperature. These alloys are frequently referred to as "non-age-hardening."

For practical purposes this property is sometimes a disadvantage because these alloys cannot be adapted so easily to the manufacturing processes as can the usual age-hardening alloys, which can be used in a more ductile and weaker state or in a stronger and less ductile state by suitable choice of thermal treatment. Aluminium-magnesium alloys, since their mechanical and forming properties are practically unchanged by thermal treatment, must be formed in the condition in which they are used in service. On the other hand, this effect allows more freedom in the choice of the heat treatment of the aluminium-magnesium alloys than is the case with the usual age-hardening aluminium alloys. For example, in alloys of the aluminium-magnesium-silicon type a choice has to be made between maximum strength or maximum corrosion-resistance, since the condition of highest strength does not correspond with that of maximum corrosion-resistance. The fact that in the aluminium-magnesium alloys the mechanical properties are practically independent of the thermal treatment enables the heat-treatment to be selected so that other properties, such as corrosion-resistance, reach optimum values. This viewpoint is the basis of the investigations described in this paper on the influence of chemical composition and thermal treatment on the intercrystalline corrosion of aluminium-magnesium alloys containing 5–9% magnesium.

As a result of the investigation, a new method of heat-treatment, termed a "stabilising treatment," is suggested in which the alloy is extremely slowly cooled from the homogenising temperature at a rate of about 50° C./hr. Corrosion tests in the laboratory and in the North Sea, as well as long-term service trials, indicate that alloys treated by this method are immune to intercrystalline-corrosion and stress-corrosion failure even at the high temperatures encountered under tropical conditions.

While cold reduction generally increases the tendency to intercrystalline corrosion, small additions of copper, manganese, chromium, and zinc have very different effects. Some of these additions, e.g., copper, reduce the stress-corrosion-resistance in the homogenised state and improve it in the aged condition. The high stress-corrosion-resistance of alloys containing small additions of chromium instead of the usual manganese is of special interest.

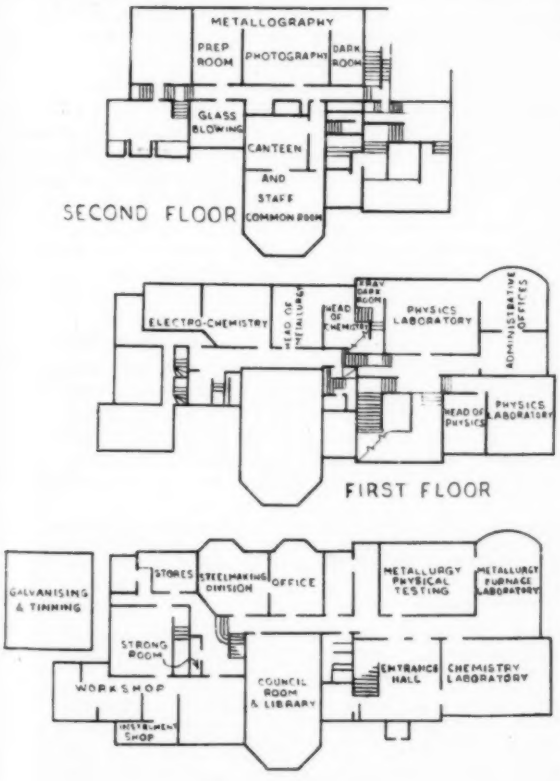
The close correlation between the thermal treatment, the micro-structure, and the susceptibility to intercrystalline corrosion and, especially, stress-corrosion of these alloys is clearly indicated.

The South Wales Laboratories at Sketty Hall

Co-operative Research Activities of the British Iron and Steel Research Association

Since the founding of the British Iron and Steel Research Association in 1945, much progress has been made in the establishment of several laboratories. The first of these are the South Wales Laboratories at Sketty Hall, Swansea, which were officially opened a few months ago to establish and maintain liaison with the iron and steel works. The work of these Laboratories is mainly concerned with inorganic coatings on steel and processes for surface preparation, but steelmaking researches are also included.

IN the past 30 years or so, control of steelmaking processes, and many of the operations involving steel manufacture, have changed from almost a pure art to a scientifically founded technology based partly on applied science and partly on accumulated experience and traditional art. Production control laboratories were established in works and the desire for knowledge concerning the underlying principles governing the diverse technical problems of the iron and steel industry led to the growth of centralised laboratories where such problems could be studied. Gradually co-operative research, to supplement industrial and university laboratories, found favour and, the Technical Department of the British Iron and Steel Federation and Joint Committees formed with the Iron and Steel



Plan of the laboratories.

Sketty Hall from the South.

Institute, together with a small, but active department in South Wales, have carried out co-operative research for many years. These organisations and committees became the nucleus of the British Iron and Steel Research Association formed in 1945. The main functions of this Association are concerned with background or objective research and with new techniques, processes, etc., of common interest. Since its foundation, several laboratories have been set up and staffed to carry out work within a particular field and to maintain liaison with works engaged on manufactures within that field. The more recent of these are established at Sketty Hall, Swansea, which are mainly concerned with inorganic coatings on steel and with processes for surface preparation, although steelmaking researches are also undertaken.

The laboratories, as indicated in the accompanying plan, are disposed on three floors, and a galvanising and tinning plant is being installed in an adjacent building. Mr. D. Luther Phillips, M.Sc., F.R.I.C., who was formerly in charge of the research work carried out at Swansea University by the South Wales Siemens Steel Association, is in charge, with a scientific staff of 18 and a total staff of 26.

The location of the various laboratories and offices is indicated on the plan. On the ground floor there is a large chemistry laboratory, furnished with all the usual equipment, including absorptiometric and electrometric titration apparatus. There is also a metallurgical furnace laboratory equipped with both gas and electric heat-treatment furnaces with a full range of temperature

Fig. 1

Fig. 2

Fig. 3

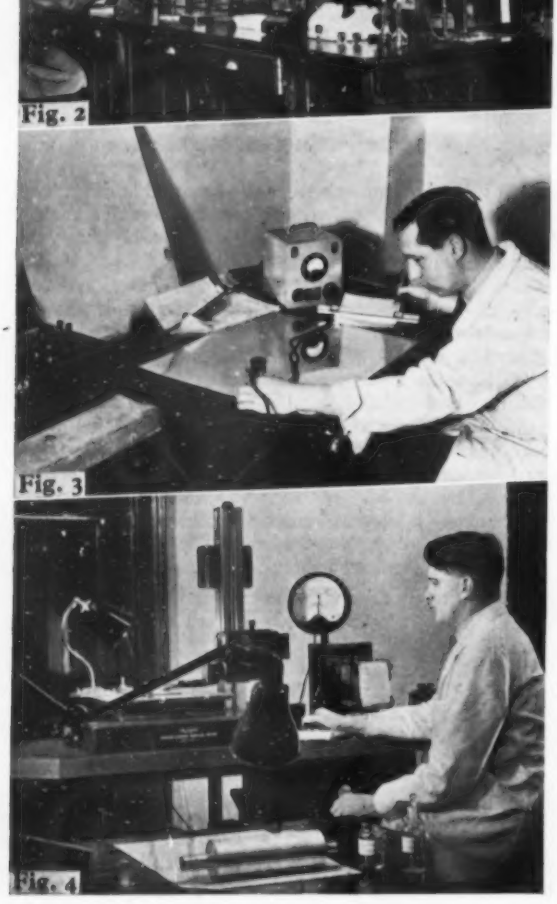


Fig. 4

measuring and recording equipment. On the same floor is the metallurgy physical testing laboratory, which has an electro-magnetic instrument for layer thickness measurement, tensile testing, Vickers hardness testing and Erichsen cupping testing machines and so on. A well-equipped workshop and instrument shop (where the whole of the galvanising plant now being installed has been made) a large office for B.I.S.R.A's Steelmaking Division and a council room and library.

On the first floor there are two physics laboratories and two for the electrochemical section. Equipment in the former includes a Talysurf machine carrying a stylus which traverses a specimen and records the roughness of the surface on a graph with an adjustable magnification of from 400 to 100,000 times. An "average meter" can then give the average roughness of the surface. It is thus possible to investigate the geometrical characteristics of surfaces with ease and rapidity. In addition to the Talysurf machine, there is X-ray diffraction equipment and a version of the Guild photometer, as well as the more usual equipment of a physics laboratory. The Guild photometer is an optical smoothness meter which operates by concentrating a beam of light on a small area of the surface to be tested, all the reflected light being collected inside a small integrating sphere. By measuring the specular reflection from the sample, in relation to the total light reflected in all directions, a very sensitive means of comparing surfaces for smoothness may be obtained.

Equipment in the electrochemical laboratories includes a 250 amperes, 3-phase rectifier, supplying direct current at voltages up to 16. Large tanks, with their relevant switch-gear, and current-and-temperature-control equipment, for sulphuric acid electrolytic pickling, alkali degreasing and chromium plating, have been installed, and provision made in these large baths for rapid warming-up, so that long delays in waiting for working conditions are unnecessary. Provision has been made for connecting up smaller vats for the disposition of zinc, tin, nickel, etc. A filtering device has been set up capable of effecting the purification of the plating solutions in the largest vats without attention. Among equipment in these laboratories, devised and constructed on the spot, is an all-glass apparatus for obtaining electrode potential-current density curves, and interrupter-potentiostat equipment of great value for the study of electro-chemical processes. This last can maintains electrode potentials at any desired value, while the interrupter section enables decay of electrode polarisation to be studied from 10^{-6} secs. onwards.

The second floor has comprehensive metallographic facilities, with separate preparation laboratory and photographic rooms. A small room equipped for glass

Fig. 1.—Section of furnace room. On the right, gas heated muffle and crucible furnaces. On the left, apparatus for studying interaction of zinc and iron, using electrically heated crucible furnace, with automatic control.

Fig. 2.—Analytical laboratory showing photo-electric absorptiometer, experimental pickling equipment and potentiometric titration outfit.

Fig. 3.—Testing laboratory, showing thickness determination of heavy tin coatings by means of electromagnetic thickness meter.

Fig. 4.—Physics laboratory showing the Talysurf instrument and, in the foreground, the roll replica technique.

blowing and the staff canteen and common room complete the accommodation on the second floor.

Work in Progress

Coatings

Approximately 75% of the work of these laboratories is concerned with coatings and is organised under the Association's Coatings Committee, of which Captain H. Leighton-Davies is chairman, to consider and investigate all matters concerning inorganic coatings on steel and processes for surfaces preparation. This involves close liaison between this laboratory and the Association's corrosion laboratory at Birmingham to avoid the possibility of overlapping. However, this danger is not likely to arrise since one is concerned with coatings application, while the other deals with utilisation.

It is generally recognised that the effect of a coating on steel, applied intentionally for protection but without regard to scientific principles, is uncertain, but many factors remain to be studied to improve the resistance of coatings and the general programme of work in progress at these laboratories falls under five heads :—

1. The steel base and its preparation.
2. The coating materials.
3. Methods of application of coatings.
4. The composite (or coated article).
5. User problems.

Each of these classes of investigation calls for close co-operation between laboratory and works, to keep industry and laboratory mutually informed of progress and practice. This means that the Association's staff at Swansea spend a considerable proportion of their time on " field " work at various works, and this healthy development is shortly to be reinforced by the appointment of a full-time officer for this purpose. Industry, moreover, is brought to the laboratory by exchange visits from works and laboratory staff, and both plating and galvanising plant are part of the laboratory's equipment. The latter, designed and built on the premises, is now being installed. The former has been in operation for some time.

The steel base and its preparation.—Particular importance is attached to the study of the surface of the steel base prior to coating, for which purpose the Talysurf instrument, the Guild photometer and the X-ray diffraction equipment are largely used. Electron-diffraction equipment is to be installed to reinforce the fundamental examination of surface structure. In this connection the various influences of the methods of steel making and intermediate stages of manufacture such as rolling, annealing and pickling are the objects of the study. In studying the effect of various degrees of cold rolling, for example, the Talysurf gives a quantitative measure of the effect on the surface, for correlation with the amount of tin deposited. Similar correlations are obtained for various pickling conditions, such as concentration, temperature, time and inhibitor variations.

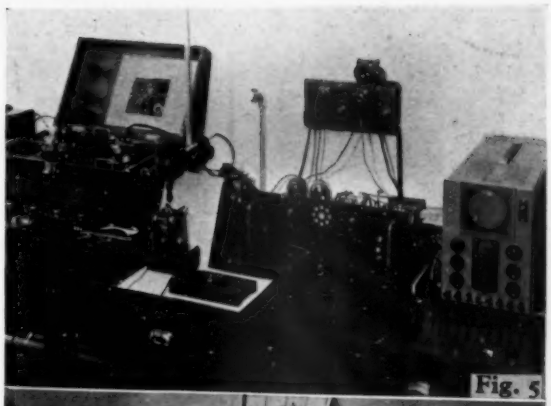


Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 5.—Section of electrochemical laboratory, showing pH meter for measuring hydrogen/ion concentration in solutions; interrupter-potentiostat equipment and cathode ray oscillograph.

Fig. 6.—Corner of workshop.

Fig. 7.—The laboratory's galvanising unit in course of installation, showing frame raised out of the pot and conveyer on the left.

Fig. 8.—Quick immersion pyrometer on demonstration at a South Wales works.

This series of projects includes study of the influence of roll surface, and a new replica technique has been developed for bringing to the Talysurf machine a record of the roughness of the rolls in a mill. Plastic "prints" are taken of portions of the roll surface, the difficult part of the technique having been to evolve a method of applying the plastic so as to reproduce faithfully the small-scale irregularities of the surface, and yet be capable of easy removal. In practice, a drop of a liquid mixture of monomeric and polymeric methyl methacrylate, containing a photochemical catalyst and an organic ester to act as a "stripping agent," is placed on the surface to be examined. A piece of Perspex sheet (1 in. × 1 in. × $\frac{1}{8}$ in.) is pressed on this drop so as to produce a thin film of liquid between the metal and the Perspex. The specimen is irradiated by an ultra-violet lamp for one hour, and at the end of this period the liquid has been set, and the replica can be readily removed. Talysurf records, taken at high magnification, of corresponding areas of metal and replicas, show that faithful reproduction is achieved, though the replicas are, of course, negatives of the metal surfaces.

The replicas are eminently suited to microscopic examination, particularly by transmitted light, as the polymerised liquid takes a faithful impression of all surface irregularities. Photomicrographs of etched metals and their replicas are indistinguishable except for slight changes of photographic contrast.

Future projects which will come under this head are concerned with the chemical characteristics of the steel surface at all stages before, during and after the coating processes. The influence of methods of manufacture, for example, of killed, semi-killed or rimming steels, of basic Bessemer and Open Hearth steels in this connection remains to be examined.

The coating materials.—The main project under this head is the study of the influence of elements added to the galvanising bath on the application, appearance and behaviour of the coating. The galvanising machine now being installed is designed to deal with sheets up to 8 in. wide and from about 20 in. upwards in length—the longer lengths being used for examining longitudinal distribution under varying conditions. By means of this machine it will be possible to examine, for instance, the influence of various elements in the zinc bath on spangle formation and rate of formation of alloy layers under conditions comparable with those of industrial sheet galvanising practice.

Methods of application of coatings.—These include problems encountered in :—

(i) Hot dipping, where some of the variables to be examined are sheet temperature and velocity, conditions at the exit rolls, method of with drawal, etc.

(ii) Electrodeposition where the value of the interrupter potentiostat is very great in studying the fundamental mechanism of deposition processes.

(iii) Spraying.

(iv) Diffusion on cementation processes.

(v) Deposition on gaseous reaction processes.

The Composite (or the coated articles).—Work is being carried out on the thickness, character and distribution of coatings. This has involved among other things, the metallographic examination of the zinc coating on galvanised sheets on a considerable scale. A zinc coating which appears to be fairly uniform, when sampled for thickness by the usual stripping method, may be found, on microscopic examination of its cross section, to fluctuate considerably over very short distances. The relatively large sample usually taken for coating determination tends to average out these "micro-irregularities" and so to conceal them. This effect is largely due to the condition of the steel surface to which the coating has been applied. This ties up, therefore, with investigations of the influence of irregularities in the steel surface on the coating process, and work along these lines is considered to be of considerable importance.

Another aspect of the work under this head is investigation into the effect of various cleaning treatments on the metal electro-deposited on cold rolled sheet. Results so far indicate that Talysurf measurements should be of great value in studying the course of electrodeposition of metallic coatings, particularly where these are very thin.

User Problems.—These may be briefly listed :—

(*a*) The effect of surface contaminants and film such as oil, soap, moisture, etc., on the receptivity of the surface to tin plate decoration lithography, etc.).

(*b*) Seam strength of soldered joints. Comparison of soldered joints on electrolytically deposited tin as against hot dipped tin.

(*c*) Investigation of methods of determining porosity; its assessment and prevention. Considerable progress has been made in evolving a new method of determination.

(*d*) The investigation of treatment of coatings to improve certain specific properties, e.g., resistance to staining, adhesion of coatings.

The Staff of the South Wales Laboratories.

Back Row : S. Jepson (Chemistry), R. Humphreys (Metallurgy), M. Evans (Steelmaking Section), R. James (Gardener), T. Canning (Chemistry), D. F. G. Thomas, B.Sc. (Metallurgy).

Middle Row : Miss Davies (Secretary), H. N. Jones, M.Sc. (Metallurgy), D. P. Moses, B.Sc. (Metallurgy), D. G. B. Thomas, B.Sc. (Physics), J. P. Riddle, B.Sc. (Physics), I. E. Thomas (Instrument Maker), I. Douglas (Steelmaking Section), W. Bullough, B.Sc., A.R.I.C. (Chemistry), A. Stephens (Mechanic's Assistant), Miss Austin (Typist).

Front Row : F. W. Salt, Ph.D. (Electrochemistry), M. R. Hopkins, M.Sc., F.Inst.P. (Head of Physics Section), M. L. Hughes, M.Sc., F.R.I.C., F.I.M., A.M.I.Chem.E. (Head of Metallurgy Section), D. Luther Phillips, M.Sc., F.R.I.C., F.I.M. (Head of Laboratories), J. Pearson, M.Sc., Ph.D., F.R.I.C. (Head of Chemistry Section), R. Toye, B.Sc., A.M.I.E.E., A.Inst.P. (Head of Steelmaking Section), L. Harold (Establishment Officer).

Two Additions to Staff since photograph was taken :
T. C. Toye, B.Sc. (Physics), D. G. Thomas (Metallurgy).

Steelworks Problems

Work other than on coatings is organised under the South Wales Steel Sub-Committee, of which Mr. J. S.

Lewis is chairman. This is the only one of B.I.S.R.A's Committees organised on a territorial rather than a functional basis. Its terms of reference are "To discuss and investigate the manufacture of steel for flat steel products."

Generally speaking, the many subjects involved in this definition are dealt with by their respective research committees throughout the B.I.S.R.A. organisation. The South Wales Committee therefore, avoids duplication of effort by assisting in the relevant projects of other committees rather than in undertaking its own. At Sketty Hall, projects undertaken on behalf of this Sub-Committee largely involve field tests on melting furnaces and works in the South Wales area. These projects are concerned with such matters as fuel distribution and gas flow in gas producers (in conjunction with the British Coal Utilisation Research Association); the effect of deterioration of ingot mould surfaces on the surface of the ingot and subsequent products; the determination of the moisture content of hot raw producer gas, and the assessment of the value of instrumentation of open hearth furnaces.

In this last connection, liaison with the various works is very close. It has extended on occasion, to mutual exchange and loans of instruments, such as roof pressure meters, and demonstration of the quick immersion thermocouple has been made at many works requesting it. A new method of determining the moisture content of hot raw producer gas is another project successfully completed, while a method of taking replicas in plaster of paris of crazed mould faces has also been successfully used.

Among the immediate objectives of all industrial research organisations are the maintenance and improvement of quality, utility and consistency, and the securing of economy in materials and method. In pursuit of these aims, the South Wales Laboratories are playing their part in Britain's economic struggle.

Hydrogen, Nitrogen and Oxygen in Ferrous Metals

Their Properties and Their Determination—Part V

By E. C. Pigott

A perspective of the main experimental and co-ordinating work carried out by chemists and metallurgists on oxygen in irons, steels and ferro-alloys was commenced in the last issue of this journal. In the present article of the series methods are discussed for the determination of oxygen in ferrous metals.

Gravimetric Aluminium Reduction Method

An alternative to the vacuum-fusion process for determining total oxygen is the aluminium reduction method of Gray and Sanders (J. Iron Steel Inst., 1938, No. 1, 348P; 1941, No. 1, 321P; 1944, No. 2, 242P; 2nd Report Oxygen Sub-Committee, 1939; 103). This consists in reducing the oxygen-bearing inclusions by heating the sample at 1,150° C. between two pieces of aluminium metal, contained in a graphite boat, either in an atmosphere of hydrogen or under reduced pressure, with subsequent acid solution of the iron-aluminium melt, and chemical determination of the alumina so formed. The method is applicable to carbon steels, including those high in sulphur or phosphorus, and to low alloy steels. The results obtained agree closely with vacuum-fusion yields, but tend perhaps, to be very slightly lower. Precautions must be taken to keep the blank yield low. The use of red-rubber tubing is not permissible, since it can lead to the formation of a film of aluminium sulphide, not readily soluble in the acid mixture employed. Tungstic acid may be removed from the final alumina residue by means of ammonium hydrate.

The method is applicable to pig irons generally, provided a suitable sample can be obtained. Certain samples can be cut with a hack-saw, but the use of a carborundum wheel gives rise to serious surface oxidation. A modification necessary with pig irons is that of treating the unignited residue with sodium carbonate and sodium citrate in order to remove hydralysed silicic acid, which would adsorb aluminium salts from the solution, so causing an excessive amount of alumina to be present in the final residue. (Taylor Austin, J. Iron Steel Inst., 1941, No. 1, 328P).

As operated by Stevenson and Speight (J. Iron Steel Inst., 1941, No. 1, 326P; 1943, No. 2, 242P), the reduced pressure method will be described in a succeeding part.

Residue Methods

Residue methods are less direct, less rapid, and less widely applicable than the vacuum-fusion process. They are still largely research methods, a single determination occupying several days, but, nevertheless, they have definite merits within their special field of application. The equipment required is much less costly and complicated than vacuum-fusion apparatus, and accurate values are obtained not only of total oxygen, but also of the individual oxides. A disadvantage of residue methods is that they do not record any oxygen-bearing gases entrapped or dissolved in the steel.

Extraction of the oxides can be effected by means of halogens or dilute mineral acids or electrolytically. In general, acid-solution methods have been superseded by the more rapid and reliable halogen methods, of which the iodine and chlorine methods are outstanding.

Eggertz' original iodine method has undergone considerable modification, notably at the hands of Cunningham and Price, Taylor-Austin, Kinzel, Egan and Crafts. Kinzel's procedure consists in dissolving the metallic matter in an ice-cold solution of ferrous iodide and

ammonium citrate, with ignition of the residue and estimation of the SiO_2, MnO, FeO and Al_2O_3 by conventional methods. The chlorine process has been developed by Oberhoffer and Wasmuht in Germany and by Colbeck, Craven and Murray in this country, where also the alcoholic iodine method of Rooney and Stapleton is applied to steels and the aqueous iodine method of Taylor-Austin is applied to cast irons.

Dickenson's procedure, which consists in extraction with cold 10% nitric acid and removal of silicic acid in 10% sodium hydroxide solution gives useful comparative results on well-deoxidized steels, in which the oxygen exists largely as silicates of manganese and aluminium. Herty modified the hydrochloric acid method devised by Kichline. Perchloric acid has been perposed as a reliable selective solvent.

Fitterer's electrolytic method of dissolving the metallic matter in a solution containing 3% of ferrous sulphate and 1% of sodium chloride was modified by Treje and Benedicks, who substituted a bromide solution, in order to reduce anodic acidity, and who inhibited the hydrolysis of iron by means of citrates; the retention of carbides is troublesome, but the method is satisfactory for silica. Their version of the method has been modified in turn by Klinger and Koch. In many cases, with the possible exception of alumina values, good agreement is secured between the total oxygen assessed from the electrolytic separation of inclusions and that obtained by vacuum fusion, but the electrolytic procedures have not so far proved as reliable as the iodine and chlorine methods.

Absorptiometric methods for analysis of the extracted residues have been devised by a number of investigators.

Alcoholic Iodine Method

The alcoholic iodine method of T. E. Rooney has undergone much development since publication of the original paper (Rooney and Stapleton; J.I.S.I., 1935, No. 1, 249), and, in the absence of large amounts of certain alloying elements, is altogether dependable. The method is based on the selective solvent action of a methyl alcohol solution of iodine, which converts the iron, iron carbide and iron phosphide into soluble iron iodine, but does not attack the various oxides, which remain insoluble and are removed by filtration, and ignited, and analysed by microchemical or spectrographic means. The utmost precision is called for in this subsequent analysis, since the amount of separated residue available is extremely small.

Most alternative iodine solvents fail to dissolve the iron iodide formed and are, therefore, unsuitable. They include acetone, trichloro-ethylene, benzene, carbon tetrachloride, and ethyl dibromide. Methyl alcohol, a fairly consistent commercial product is preferable to ethyl alcohol. It is necessary to deoxidize the alcoholic iodine solution, and this is done by passing dry nitrogen which serves equally as well as hydrogen.

The method is ordinarily applicable to steels of low or medium carbon content not containing appreciable amounts of elements which form stable carbides—e.g., chromium, vanadium, titanium and molybdenum, or which, like titanium, form insoluble iodides. It is not ordinarily applicable to cast irons and pig irons, largely owing to the interference set up by the considerable amounts of carbide and phosphide present; phosphorus and iron oxides, which are not present originally, contaminate the residue to an extent largely dependent upon the iron carbide content of the metal. Phosphoric pig—and cast-irons particularly, in which the phosphide inclusions are associated with titanium, give residues extremely high in iron oxide and phosphorus pentoxide, no matter how long the solution is subjected to agitation. The residue may contain also undissolved iron phosphide.

Decomposition of the steel sample is complete if 5 gm. of iodine are used per gramme of steel. Slightly smaller amounts of iodine are permissible, but retard the reaction rate, and in practice a somewhat greater preponderance is used.

Solid disc-shaped samples of thin section and weighing about 8–12 gm. are used. Millings have been found to be unsuitable owing to the extent of surface oxide films. Normally, decomposition is carried out at 60°–65° C. in order to ensure the complete breakdown of sulphides. Rimming steels are dealt with at a lower temperature, because the oxides of iron and manganese contained in them are such that they become partially soluble at 33° C. and above. The water-content of the methyl alcohol must not exceed 0·2%. Higher contents cause an increase in the percentage of FeO and P_2O_5 in the residue. Hydrometers serve to indicate the amount of water present and when, as often happens, a higher content is indicated, it is necessary to distil over calcium. A single distillation can reduce the water content from, typically, 0·25% to about 0·18%; further distillation is seldom advantageous and actually is liable to increase the amount of water.

In the early stages of development of the method various forms of agitation were investigated—viz., stirring, churning at room temperature, percolating, and boiling. The churning and boiling methods proved very lengthy, and the percolation method was not applicable to massive-type samples. For these and similar reasons the Stirring Method, as devised at the N.P.L., became standard. In some cases, it is an advantage to prolong the stirring time beyond the stage of complete decomposition of the sample.

Effect of Carbon

Carbon, especially that present in the steel in the non-pearlitic condition is left largely in the elemental form as a fine voluminous suspension, which, possesses powerful and adverse adsorptive properties. It adsorbs iron and phosphorus oxides as well as aluminium and traces of silicon. Fortunately such contamination can be reduced very considerably by a simple after-treatment. The adsorptive effect is at a maximum where the contents of carbon and adsorbable constituents are high; should the carbon content exceed about 0·6%, the residue contains an excessive amount of iron, yielding a high value for total oxygen, and even with somewhat lower carbon contents the residues require suitable after-treatment in order to ensure efficient washing. The maximum permissible carbon content is limited also by the low solubility of iron carbide in the iodine solution; where the carbon content is high, the iron carbide does not completely decompose.

Effect of Aluminium

In addition to the alumina constituent, aluminium-killed and aluminium-alloyed steels may contain other aluminium compounds, such as the carbide and nitride. The decomposition both of carbide and nitride is slow and incomplete and they are partially retained in the residue, while elemental aluminium is adsorbed by the free carbon formed by the breakdown of the iron carbide. Treatment with 10% ammonium tartrate is effective in

removing the carbon-adsorbed aluminium. It is still necessary to distinguish between true inclusion alumina and that derived from the carbide and nitride, and the customary method is to digest the gently ignited residue in hot 1 : 1 sulphuric acid, which dissolves the derived alumina, but not the inclusion alumina.

Aluminium carbide is decomposed by water or acids, while aluminium nitride is soluble in alkalis, and in practice the ignition is preceded by treatment with dilute sodium carbonate solution.

Effect of Phosphorus

When the oxygen content (including surface film oxygen) is very low and the alcoholic iodine is adequately deoxidized, normal phosphorus contents do not interfere. Speight has shown that phosphorus is adsorbed by the free carbon formed by the decomposition of iron carbide, but that that so adsorbed can be substantially reduced by treatment of the residue with boiling 10% ammonium tartrate solution, which also removes iron, aluminium and silicon contamination. Given equal carbon contents, the extent of P_2O_5 contamination depends more or less on the phosphorus content of the steel, but is greater in the case of steels than in that of normalised steels. The presence of water in the methyl alcohol increases the amount of phosphorus remaining in the residue. Part of the phosphide is broken down by the alcoholic iodine, yielding soluble iron iodide and free phosphorus, which, also, dissolves. The dissolved phosphorus is then converted to phosphate, either by the weakly ionizing properties of the alcohol or, especially, by interaction with a trace of water. Ultimately, the residue contains phosphate, possibly mixed with iron phosphide.

Neither extra washing with alcohol nor a second treatment with alcoholic iodine serves to remove much of the carbon-adsorbed phosphorus, or for that matter, any of the contaminants, but, as in other cases of adsorption analysis, separation of most of the contaminating phosphorus can be effected by after treatment with a solution of entirely different chemical characteristics. Solutions of sodium carbonate, ammonium tartrate and ammonium citrate have been applied with success. It is altogether safe to assume that the P_2O_5 in the residue is not derived from oxygen-bearing inclusions. The small quantities that separate must be determined, and a correction made, otherwise high results will be recorded for alumina and for total oxygen.

Effect of Sulphur

Sulphides are decomposed upon solution of the simple, and elemental sulphur is precipitated. Decomposition is markedly influenced by the maximum extraction temperature attained. In the decomposition of steels other than those of normally low sulphur content it is necessary sufficiently to prolong the stirring time. Normally, no sulphides are present in the residue, and the free sulphur is removed by ignition and can be disregarded. There are exceptions: low-carbon high-oxygen steels such as are frequently made by the Basic-Bessemer process, are liable to leave a residue containing undecomposed manganese sulphide, while there is also a possibility of the retention of the titanium-sulphur compounds present in high-titanium steels, especially those of high phosphorus content.

Effect of Nickel and Copper

Both nickel and copper dissolve completely and are without interference.

Effect of Molybdenum

Molybdenum in steels is not present in combination with oxygen; a small proportion of the element remains undissolved, but this volatilises during the ignition. Interference may arise if the molybdenum content is high.

Effect of Chromium

A small proportion of the chromium content is retained in the residue, but provided the chromium content is not too high, this residual amount can be considered in the course of analysis, and then ignored, since chromium does not occur as an oxygen-bearing constituent.

Effect of Titanium and Vanadium

Titanium and vanadium, strong carbide-forming elements, interfere seriously. Usually, more than half the content of each remains undissolved. Titanium iodide is not very soluble, while titanium carbide is not attacked at all and remains wholly in the ignited residue as the dioxide. It follows that the determination of true inclusion—titanium is scarcely possible by means of this method.

The Chlorine Method

The chlorine method of Colbeck, Craven and Murray was first described in 1936. Since that time, the method has undergone modification, and its range of application has been extended. The process is based on the volatility of the chlorides of iron, silicon, manganese, etc., at a temperature at which the oxide inclusions are not attacked. Subsequent analysis yields the amounts of the individual oxides. As with all residue methods this analysis requires skill of a high order; it is also time-consuming, so that the method is used primarily for research purposes.

The method is applicable to killed plain carbon steels and to alloy steels containing amounts up to at least 0·5% of chromium, molybdenum, nickel, titanium or copper and 1·5% of manganese. Results obtained on these materials are reproducible and agree closely with vacuum-fusion values. Recently, the method has been made to serve for certain of the more richly alloyed steels.

Rimming steels do not respond to the method, for the inclusions present in them consist largely of FeO and MnO (unlinked with appreciable amounts of SiO_2 or Al_2O_3, and it would appear that these are subject to attack by the chlorine, since the results obtained are very low. Nor is the method applicable to grey cast irons and pig irons. In the presence of graphite, large quantities of manganese are retained in the residue. When applied to white irons strictly free from graphite, the process is capable of giving very satisfactory results.

In contrast with the alcoholic iodine method, the chlorine process is not subject to interference from aluminium carbide and nitride, or from moderate amounts of carbide-stabilizing elements, or from phosphorus. Even when existing together, large amounts of phosphorus and sulphur do not affect the results. With high phosphorus contents of the order of 0·1%, it may be found that 0·002% or so has been retained in the residue, but such traces can readily be determined.

The chlorine used is that prepared electrolytically and purified by a liquefaction process; 1 70-lb. cylinder serves for several hundred estimations. Under the conditions devised, the difficulties sometimes apt to discourage the use of chlorine do not arise. Normally, for all practical purposes, the chlorine is oxygen-free. It is led through drying-towers, the rate of flow being about 15 litres per hour. Samples of 10–15 gm. are taken. Solid test-pieces are used, partly because of the importance of restricting the extent of surface films, and partly because the sample must be sufficiently massive to ensure full control over the exothermic reaction between the iron and the chlorine. If drillings were used, the initial rate of attack and of heat-evolution would be much too rapid. Massive samples not only react more slowly initially, but also effectively absorb the heat evolved without causing intense local heating. The standard chlorinating temperature is 350° C., and at this temperature a Pyrex glass reaction tube serves admirably. Before commencing work on a fresh series of steels, or after alterations of any kind, or when a fresh cylinder of chlorine is used, it is necessary to carry out a preliminary test on a standard steel of known oxygen content as a means of detecting any defects in the equipment.

At 350° C. chromic chloride is non-volatile in the chlorine stream, and as it is insoluble in water it is retained in the residue and subsequently ignited to the sesquioxide. This oxygen in combination with chromium in the ignited residue is not reckoned as part of the total oxygen in the steel. At 500° C., a temperature at which a transparent-silica reaction tube becomes preferable, low contents of chromium are volatilised completely. If, however, the chromium content is high the steel is far more resistant to the chlorine attack, and the volatilisation is slow and incomplete.

Such steels may be dealt with by reducing the water insoluble chromic chloride to water-soluble chromous chloride and by allowing for any unattacked metal.

The reducing agent used must have no solvent effect on the oxides present in the residue; a warm dilute solution of stannous chloride is suitable, especially in the form of a 5% ammonium citrate—2% stannous chloride solution adjusted to pH7 with sodium hydroxide. With well-killed steels containing inclusions of a balanced character, this increase of chlorination temperature to 500° C. effects little alteration in the composition and quality of the inclusions. The silica and alumina fractions do not begin to suffer attack until a temperature of 700° C. is reached. Manganous oxide remains constant up to 500° C.; it is attacked rapidly, however, at 700° C. Uncombined ferrous oxide would suffer attack at temperatures over 350° C.

It has been established that washing the residue with water in order to free it from chlorides does not take any silica into solution. In the analysis of the residue, photometric procedure is advantageous. In one recommended scheme, the first step is that of oxidizing both residue and filter with perchoric acid. The silica is then collected on a filter, ignited and weighed. Iron and titanium (also any vanadium) in the filtrate are separated by means of cupferron and chloroform as described by Breman et al (J. Ass. Off. Agric. Chemists, 1937, 20, 635), ignited to oxides and each determined colorimetrically. Aluminium, chromium, phosphorus, and manganese in the aqueous extract are determined by direct colorimetric methods. Suitable conditions for applying the aluminon reaction are those evolved by Lampitt and Sylvester (*Analyst*, 1932, 57, 418) and Speight (Third Report of the Oxygen Sub-Committee), while any traces of phosphorous respond well to the colorimetric method of Wilkinson (*J. Soc. Chem. Ind.*, 1938, 57, 292).

To be continued.

Mond Nickel Fellowship

THE Mond Nickel Fellowships Committee have announced the award of two Fellowships for 1947. They have been made to:—

MR. L. H. WALKER, Reynolds Tube Co. Ltd., to study the organisation and economics of light alloy production and the methods of application of group industrial research.

MR. D. R. G. DAVIES, Richard Thomas and Baldwins Ltd., to study the application of statistical methods to plant metallurgical and management problems, with particular reference to the steel industry.

The Committee now invite applications for the award of Mond Nickel Fellowships for the year 1948. Awards will be made to selected applicants of British nationality, educated to University degree or similar standard, though not necessarily qualified in metallurgy, who wish to undergo a course of training in industrial establishments. They will normally take the form of travelling Fellowships; awards for training at Universities may be made in special circumstances. There are no age limits, though awards will seldom be given to persons over 35 years of age. Each Fellowship will occupy one full working year. The Committee hope to award up to five Fellowships each year, of an average value of £750 each.

Mond Nickel Fellowships will be awarded in furtherance of the following objects:—

(*a*) To allow selected persons to pursue such training as will make them better capable of applying the results of research to the problems and processes of the British metallurgical and metal using industries.

(*b*) To increase the number of persons who will be competent to appreciate the technological significance of research and its results.

(*c*) To assist persons with qualifications in metallurgy to obtain additional training helpful in enabling them ultimately to assume executive and administrative positions.

(*d*) To provide training facilities whereby persons qualified in sciences other than metallurgy may be attracted into the metallurgical field.

Applicants will be required to state the programme of training in respect of which they are applying for an award, as well as particulars of their education, qualifications and previous career. Full particulars and the necessary forms of application can be obtained from the Secretary, Mond Nickel Fellowships Committee, 4, Grosvenor Gardens, London, S.W.1. Completed application forms will be required to reach the Secretary of the Committee not later than June 1st, 1948.

The Cleaning of Metals

Part V—Mechanical Cleaning Machines

By. R. Groves

For the cleaning of many metal parts the simplest forms of cleaning apparatus will suffice, but for the majority of components, machines, specially designed for the operations involved, are needed to deal with the work economically and efficiently, and in this concluding article of the series, brief reference is made to cleaning equipment.

WHEN only a few parts have to be cleaned, a simple tank will probably suffice, but it is essential that this should be big enough to deal with the dimensions and quantity of parts without becoming too full, while its design must also be correct. The disadvantage of allowing the tank to become too full because of inadequate size is that dirt accumulates quickly and prevents the solution used from doing its work to the best advantage. The tank should have a good depth, because this gives space and opportunity for sediment to collect on the tank floor, well away from the actual cleaning. Furthermore, if the solution in the tank is agitated, the depth renders possible movement of the parts in the solution without them becoming uncovered.

The floor of the tank should be given an inclination to the horizontal, with the bottom end opposite to or away from the heating coil or burner. Where the tank is of large dimensions, it is a good plan to provide a sump to receive and collect the sludge. The pipe for draining the solution from the tank is best located a few inches above the tank floor at its lowest level, particularly if the sludge is likely to be heavy or there is difficulty in avoiding silting up of the piping because of its small bore or intricate arrangement. This enables the contents of the tank to be drained away without touching the sediment, which can be cleaned out and removed later. On the other hand, if it is unlikely that the drainage system will be clogged, the drain can be located at the very bottom of the tank, so that by use of water pressure the light silt as well as the solution can be removed through the same vent.

The tank itself is normally of steel construction, though if there is any likelihood that the solution used will attack the steel, other materials such as wood, steel lined with lead or rubber, porcelain, stone, or stainless steel may be used.

The heating of the tank can be carried out by the following methods: (*a*) a steam coil of suitable heating surface immersed in the solution; (*b*) a steam jet heater pumping in live steam; (*c*) a gas burner in a convex chamber set off centre; (*d*) simple gas pipe burners; (*e*) a gas burner with a suitable coil immersed in the solution; (*f*) an electric resistance immersion heater; (*g*) strip electric heaters set below the floor of the tank; (*h*) an oil burner immersed in the solution and contained in a chamber; (*i*) an oil combustion chamber set underneath the tank. Of these methods, (*a*), (*b*), (*e*), (*f*) and (*h*) are the cheaper and better.

There are also various means of agitating the tank solution. First, is the simple agitation shield operating in conjunction with a steam coil. Second, a double agitation shield with an ebullition port, again operating with a steam coil. Third, agitation by the steam jet heater; fourth, agitation by means of a noiseless circulator heater; fifth, simple agitation of the work itself by hand; sixth, agitation by compressed air. Seventh is agitation by means of a small propeller; eighth, agitation by a centrifugal circulating pump; and ninth, mechanical agitation by means of an air hoist. It should be noted that whatever method is employed, it is vital that the solution should be brought into complete contact with the parts, as should the rinsing water.

A certain proportion of the grease and other foreign matter cleaned from the work will float on the surface of the solution, and must be removed before the parts are withdrawn, or they will once again adhere to the work surface. The various means of removing this scum are: (*a*) a permanent interior installation consisting of a sheet metal trough bent and welded along the back of the tank and placed below the level of the solution in the tank. The scum can then be collected in this; (*b*) a removable skimming trough, bent to the right shape and hooked over the edge of the tank. It has strap handles, and a bar weight sinks it when empty; (*c*) an external dam which does not interfere with the parts being cleaned, the side of the tank being slotted to receive this; (*d*) a means of drawing off the fouled solution. In this a full dam allows of separation of the floating scum from the clean solution, the fouled solution being drawn off through a tap in the side of the tank and at the top.

Ventilation of the tank to eliminate noxious or unpleasant vapours is essential, and a useful way of simplifying the cleaning of the sludge from the floor of the tank is to provide the tank with a false bottom. In this arrangement, lifting chains, which lie over the tank ends when not in use, suspend the tray-like false bottom in the solution. The tray is of wire mesh or of perforated sheet, and can collect up anything that has fallen into the tank. Below this false bottom is the real solid bottom in which the sludge accumulates.

More Elaborate Equipment

Turning now to those operations that call for something more elaborate than the simple tank, one may consider first a machine of value in applying the emulso-spray method. This comprises a pair of tanks provided with covers and having spray nozzles to give complete wetting and penetration of the parts with both the solution and the rinsing water delivered under pressure. As this type of cleaner operates at normal temperature, heating of the tank is not necessary. The parts are suspended in the washing tank by means of a rotating hanger suspended through a ball bearing swivel from a bracket at the top of the tank. The moment the spray impinges upon the parts, the bracket rotates, so

that every surface is wetted in both washing and rinsing. A machine of this type can be used where the output required is too great for the simple tank, but not enough to warrant buying an expensive washing machine.

Mechanical Washers

This brings us to the mechanical washers, which comprise a spray housing or an immersion tank having incorporated in its design proper means of handling and moving the parts to be cleaned, as well as of heating, straining and spraying solutions over the parts, and also suitable contrivances for drying and possibly cooling the parts at the end of the operation. This does not necessarily exhaust the functions that can be incorporated, but it covers the essentials, to which must also be added where necessary, the heating, agitation, cleaning and skimming devices.

The first requirement in choosing a washing machine is to ensure that the kind, dimensions, number and position of the nozzles, where the machine is of the spray type, are such that the solution is supplied to the parts at the correct pressure and volume, and without leaving areas untouched. For external surfaces it is usual to employ a flat spray, the round spray being reserved for work in which there are pockets or slots.

The pressure and capacity have to be carefully ascertained in advance, and the right kind and dimension of pump specified. The open impeller type is the more usual. It is most important that there should be no waste of solution through leaking, and this may involve special glands and packings.

Equally vital is calculation of the correct time and rate of passage through the various sections of the machine devoted to the particular operations, as if this is erroneous, the entire machine may be wrongly designed. As earlier indicated, alkaline foaming cleaners must not be employed in washing machines because of overflowing and other troubles certain to be caused. Suitable alkaline cleaners that do not foam are obtainable.

The cleaning compound must be perfectly soluble or it will choke the nozzles and clog the conveyor, etc. Alkaline cleaners in which are colloidal silicates incapable of solution must not be used.

Alkaline cleaners can be used in washing machines at a considerably lower concentration than in tanks, the difference being from 75 to 85%. Heavy cleaning operations with suitable alkaline cleaners necessitate a complete change of solution weekly or even every four days, otherwise the solution will not be effective. Tanks and pumping lines must be completely flushed out at each charging with new solution.

Washing machines are broadly divisible into continuous and non-continuous, of which the former are the more often used. The continuous conveyers can be sub-divided into straight, revolving, and machines that combine both straight and revolving. In the continuous or conveyer machines, the parts travel past successive points at which the particular treatment required is given, and as it is the parts that travel, the machine can be constructed so as to cope with the precise number of operations demanded. Moreover, machines of this type are readily built to link up with already operating handling systems, so that the parts can be automatically dealt with as part of a production line.

The non-continuous machine is sub-divisible into tunnel and cabinet types. In both, the parts remain stationary and are sprayed or rinsed from tanks of solution suitably placed. This limits the number of treatments that can be given, and in effect the non-continuous machines are usually confined to washing and rinsing, though it is possible to extend the range of operations to some extent. The work is sometimes carried from a pre-cleaning tank to a tunnel or cabinet for washing and rinsing, but this only means that the parts are dipped into the tank, removed, and then transferred to the later stages.

For this reason, the non-continuous machine is normally charged and discharged by hand, though some machines do introduce simple conveying means for location and withdrawal of the parts.

It is impossible in this article to discuss the different machines in detail, and the reader desirous of studying the various equipments available should consult the catalogues of the makers. In particular, the writer suggests that both English and American literature on the subject be studied, as only thus can a complete survey of the subject be made.

Electrocleaning Tanks

Electrocleaning tanks are important, and a few notes on these may fitly conclude this series. The solution employed is nearly always alkaline and of special type. Pre-cleaning is essential for the most successful results. The parts usually form one electrode in the solution, through which a D.C. current passes. Hydrogen is thus evolved at the cathode and oxygen at the anode and the hydrogen in cathodic cleaning prevents staining or corrosion of the surface of the parts. Much dirt is eliminated by the current action, while the hydrogen works under the solid dirt grains and detaches them. The distance between anodes and parts must be carefully calculated to provide uniform distribution of the current, especially if the parts are of complicated form. A potential of 6 to 8 volts. with a current density of 20 to 40 amps. per sq. ft. of surface to be cleaned is probably the most effective. The solution should be heated, because this minimises resistance to the current flow and quickens the cleaning. The cathode is best made of a non-ferrous alloy because it is less liable to stain and corrode. Both iron and steel can be electrolytically cleaned by both cathodic and anodic methods, as long as certain salts liable to corrode them are omitted from the alkaline solution. Brass and copper are also being cleaned by this process, and although a little staining is experienced, this is quickly removed by a brief immersion in dilute acid. The slight etching from anodic cleaning results in a superior bonded plate.

It is also interesting to note that some work is first being made the cathode for from 60 to 120 secs., then the anode for an equal period, and finally made the cathode again, for the same length of time. When the anodic method is employed, the current densities used should be from 50 to 100 amps. per sq. ft.

The alkaline cleaner used in this process must be a good wetting agent, which means that dirt will be cleaned off faster, so economising in current. The cleaner must be capable of resisting acid and hard water, and must not create undesirable scums by reason of drag-over. It should, moreover, be highly free rinsing so that it does not spoil the following solutions, and lastly, it must be able to carry the high current density desired.

Much more could, of course, be written on this subject, but sufficient has been given to indicate the importance of care in choosing the right process, and with it, the proper equipment.

Correspondence

Control of Gas Content in Bronzes

The Editor, METALLURGIA.

Sir,

We have read with great interest the work described by Dr. Pell-Walpole in your January issue on the control of gas content during the melting of phosphor bronzes, gun metals and leaded bronzes. There are, however, several points on which we are not very clear and would appreciate Dr. Pell-Walpole's comments.

In *Metals Technology*, 1947, Mr. Philips described a study on the solubility of gases in copper and the effect of soluble gases and other impurities in solid copper. According to his studies we understand that molecular hydrogen is practically insoluble in copper and probably in copper-base alloys, whereas atomic hydrogen (hydrogen in *statu nascendi*) is very soluble. It seems that the hydrogen atoms are very much smaller than the molecules and very easily penetrate the lattice of metal molecules even in the solid phase. If any hydrogen bubbles should form, the gas atoms would combine to molecules, and the bubble would be unable to penetrate through the metal.

Pure copper solidifies at 1,083° C., but the alloys have a wider range of solidification and it may be that hydrogen gas bubbles form during slow solidification and consequently porosity in alloys due to hydrogen is much more likely than in copper. I wonder if this conception is right.

Standard copper contains about 0·0025% sulphur. During solidification a eutectic of copper sulphide and copper is formed and also a eutectic of cuprous oxide and copper. There is a reaction between sulphur and oxygen and sulphur dioxide is formed causing porosity. Could it be that a certain amount of porosity in copper-base alloys is caused by sulphur?

The use of cupric oxide in the flux would oxidise also the sulphur in copper and the sulphur dioxide would escape. It also may be that sulphur in excess of the said 0·0025% causes no porosity, the standard residue amount of sulphur being harmless.

Comments on our views would be appreciated.

Yours very truly,

Outokumpu Oy, Metal Works, Pori. K. I. LEVANTO, Works Manager.

February 4th 1948.

The Editor, METALLURGIA.

Sir,

Mr. Levanto's comments on the relative effects of molecular and of atomic hydrogen are very important in relation to porosity in copper and bronzes. It is now generally established that hydrogen is soluble in copper only in the atomic state. This explains why steam or a substance containing chemically combined water may be a more serious cause of porosity in copper or bronze, than is an atmosphere containing molecular hydrogen. Water vapour in contact with the molten metal reacts rapidly to form atomic hydrogen and an oxide

$$H_2O + Me \rightleftharpoons MeO + 2H$$

The atomic hydrogen dissolves and diffuses rapidly into the metal.

Molecular hydrogen in contact with the molten metal must dissociate before it can dissolve in the metal and this may be a relatively slow process:—

$$H_2 (+ Cu) \rightarrow 2H \text{ (dissolves in metal)}.$$

The solubility of gases in metals is much greater in the liquid than in the solid state, hence during solidification, most of the dissolved hydrogen is ejected, and this re-associates to form molecular hydrogen which may become entrapped in the solidifying metal to cause porosity. In pure copper or an alloy having a very short freezing range, much of the evolved gas which is formed at the solid/liquid interface may be able to escape by bubbling through the liquid into the air above. In an alloy having a long freezing, e.g., tin-bronze, there is no sharp line of demarcation between solid and liquid, but a pasty zone in which both phases co-exist. Under these conditions of solidification, it is much more difficult for the evolved gas to escape since the bubbles tend to become mechanically entrained by the skeleton crystal network. This accounts in part for the greater sensitivity of bronzes to hydrogen porosity as compared with pure copper, but there is another important factor, viz., the oxygen contents of the respective materials.

In pure copper, hydrogen can re-act with any dissolved Cu_2O forming steam which is entrapped at the crystal boundaries. This form of steam porosity can occur with much lower hydrogen contents than are required to cause appreciable hydrogen porosity, and the former is a much commoner source of trouble than the latter in commercial coppers. A similar reaction can occur in the plain tin bronzes:—

$$SnO_2 + 4H \rightarrow 2H_2O + Sn \text{ (dissolved in bronze)}$$

but in the normal bronzes of commerce, which generally contain either zinc or phosphorus, the oxygen solubility is very low, so that steam reaction porosity occurs much less extensively.

Sulphur dioxide reacts with copper according to the equation

$$SO_2 + 6\ Cu \rightleftharpoons Cu_2S + 2Cu_2O$$

The reaction is reversible so that sulphur and oxygen together can cause severe porosity in copper as is well known.

In bronze the reaction becomes

$$SO_2 + (2Cu + Sn) \rightleftharpoons Cu_2S + SnO_2$$

This reaction occurs from left to right at ordinary temperatures occurring in casting so that no porosity is formed. At high temperatures, however, the reaction is reversible and the presence of sulphur and oxygen may then cause porosity due to the evolution of SO_2 which may become entrapped. The type of porosity formed, however, is relatively harmless to mechanical properties since it consists of small spherical cavities associated with the $(\alpha + \delta)$ constituent.

Yours, etc.

Department of Metallurgy, University of Birmingham. W. T. PELL-WALPOLE.

February 17th, 1948.

THE Electric Furnace Co. Ltd., of Weybridge, inform us that the Ohio Crankshaft Co. of Cleveland, Ohio, owners of the Tocco Process, have announced that they have purchased the induction heating division of the Budd Co., Detroit. The Electric Furnace Co. Ltd., who have the exclusive right in most of the British Empire and Western Europe to the Tocco patents, and who co-operate technically on all forms of high frequency heating with the Ohio Crankshaft Co., will accordingly now have the benefit of the Budd developments. These are chiefly connected with internal induction hardening and heating for forging.

Experience with Photometric Procedures for the Determination of Metals

By H. Cox, A.M.C.T., A.R.I.C.

Chemical Laboratory, Research Department, Metropolitan-Vickers Electrical Co., Ltd.

The application of photometric technique over a period of almost four years and some of the more important results derived from its use are surveyed. Various problems associated with the employment of photometric procedures are discussed, and certain improvements in operation are suggested. A method for the determination of chromium in steel eliminating the use of silver nitrate as a catalyst, and a composite method for the determinations of chromium, molybdenum and iron in welding fluxes are also described.

PHOTOMETRIC technique, although of comparatively recent introduction, continues to find a widening application, particularly in metallurgical analysis, and extensive work has been carried out by investigators in this and other countries on the development and application of this technique. The procedure is one which is often readily adaptable to the production of composite schemes of analysis, and examples of these have been included in a number of publications, for example, those by Vaughan[1, 2] and Haywood and Wood.[3] A considerable amount of literature has accumulated in the last few years, and two useful bibliographies have been compiled by Pigott[4] and Stillman[5].

Photometric or absorptiometric methods enable the attainment of a standard of accuracy and precision unobtainable by the older methods of visual colour comparison which, moreover, are seriously restricted in range, being confined generally to the determination of amounts below 1%. The old-established procedures of chemical analysis are frequently of a tedious character, involving a multiplicity of operations, and making considerable demands on the experience, skill and time of the chemist. This becomes very evident when the work is concerned with the determination of metals in the same analytical group. Photometric technique does not necessarily offer a simple solution of the more involved types of analysis or mean less careful manipulation in the preparation and treatment of the sample prior to extinction measurement. Analytical procedure, however, can often be simplified to a marked degree, and as a result appreciable time-saving is effected in many cases. This is an advantage in all industrial laboratories which control materials and manufacturing processes, and whose function includes speedy analysis, whilst yet maintaining a degree of accuracy in conformity with the requirements of the processes, etc., in which the materials are involved. Since in most laboratories a major proportion of this type of analytical work is frequently carried out by the younger staff, the manipulation of new methods developed by senior personnel must not be beyond the capabilities of the younger assistants, whose fundamental knowledge and experience is limited by reason of their age.

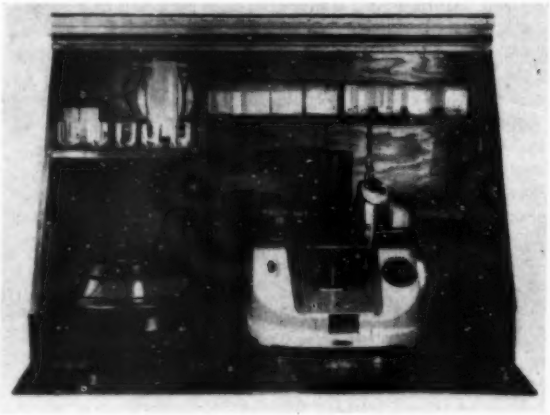


Fig. 1.—The Spekker instrument with additional fittings as used by the author.

Controlling Factors

Visual colorimetric methods have been used for many years, and it might be imagined that their adaptation to photometric measurement would be comparatively simple. However, it is very evident from the author's experience that before such adaptation can be made, the conditions required for the production of colour complexes of intensity and stability suitable for photometric measurement must be investigated in the most careful manner, and the results critically scrutinised. It has been found in a number of cases that published methods have required investigation and modification when taken as the bases for the development of procedures, and Stross[6] would appear to have had similar experience.

In a large number of visual comparisons the coloured solution and blank are adjusted to similar depths of colour by the addition of a standard solution to the blank, and assuming that identical treatment has been applied throughout to each solution, satisfactory results are obtained. When using the photometric method, a calibration graph is established under defined conditions, and extinction measurements are carried out subsequently on samples received for analysis. It is obvious

1 Vaughan, E. J., "The Use of the Spekker Photo-electric Absorptiometer in Metallurgical Analysis," Royal Institute of Chemistry Monograph, 1941.
2 Vaughan, E. J., "Further Advances in the Use of the Spekker Photo-electric Absorptiometer in Metallurgical Analysis," Royal Institute of Chemistry Monograph, 1942.
3 Haywood, F. W., and Wood, A. A. R., "Metallurgical Analysis by means of the Spekker Photo-electric Absorptiometer," Hilger, Ltd., 1944.
4 Pigott, E. C., "Photo-electric Colorimetry," *Iron and Steel*, 1944, **17**, 364.
5 Stillman, J. W., "Bibliography of Photo-electric Spectrophotometric Methods of Analysis for Inorganic Ions," Symposium on Analytical Colorimetry and Photometry, page 740, Amer. Soc. for Testing Materials, 1944.
6 Stross, W., "Rapid Photometric Determination of Copper, Nickel, Manganese and Iron in Aluminium Alloys," *Metallurgia*, 1945, **32**, 257.

that a departure from closely controlled conditions in the former case of visual comparison may not be serious provided that it applies to both solutions, whereas a departure in the latter case (i.e., applied to the sample only) cannot be tolerated. Turbidity which may be accommodated by the eye in visual methods will prove fatal to accuracy when present under photometric measurement.

The necessity, prior to using a method, of establishing the limiting conditions and tolerances upon which colour density and stability are dependent is exemplified by numerous instances, and this necessity is dealt with in some detail in a recent publication of the A.S.T.M.[7] Thus, the determination of iron by the thiocyanate reaction requires selection of the type of acid, and close control of acid concentration and quantities of added reagent, whilst the conditions controlling the production of a stable oxythiocyanate complex for the determination of molybdenum[8] provides another example of the need for close specification of procedure. A further illustration is provided by the determination of copper by means of the diethyldithiocarbamate complex, where the pH value[9] is of great importance. Difficulties encountered in this laboratory in the development of the copper complex and traced to variations in the specific gravity of the alleged 0·88 sp. gr. ammonia were overcome by modifying the quantities used. This point has been the subject of recommendations by Phillips and Edwards,[10] and later confirmed by Stross.[6] Recent work in this laboratory on the photometric determination of small amounts of copper in magnesium metal has shown that the use of hydrogen peroxide[3] causes fading of the colour, and at a rate increasing with the concentration of hydrogen peroxide. The author has found nitric acid to be a satisfactory substitute for this reagent.

Examples of Application

It is proposed in this paper to review almost four years' experience with the application of photometric analytical procedures in the laboratory with which the author is associated, contrasting these with the methods previously employed. The Spekker Photoelectric Absorptiometer has been used exclusively throughout this work.

A very large and increasing volume of work was required for the control of the manufacture of sintered metal carbides, and the use of photometric technique was applied first in this connection, with the primary object of expediting the supply of analytical data to the manufacturing departments, and also with a view to enabling the existing personnel to carry the increasing load. A composite method[11] was developed by which determinations of cobalt and titanium (both to a maximum of 15%), and iron (1% maximum) were carried out on one sample weighing, these three metals plus carbon constituting the control analysis of the material. The anticipated results with respect to time-saving and capacity of personnel to handle a greater volume of work were fully realised, two assistants being able to carry out 1,000 determinations per month. Cobalt had previously been determined gravimetrically by the α-nitroso-β-naphthol method, and later, with a certain amount of simplification, by electrolysis. Titanium had been determined gravimetrically utilising the cupferron precipitation, and iron gravimetrically as oxide. These three determinations, in the aggregate, demanded considerable manipulative work and were comparatively lengthy.

The introduction of tantalum into certain types o sintered carbides presented some difficulty, as the separation of tantalum and titanium is a time-consuming and involved procedure necessitating skilful and experienced handling. The isolation of the two metals together is relatively simple; therefore the combined titanium-tantalum precipitate was taken into solution and the titanium determined photometrically, the tantalum percentage being obtained by difference. A degree of accuracy, sufficient for control purposes was obtained, such as would be unattainable by visual comparison, because of the high percentage of titanium relative to the tantalum present in the carbide.

The determination of molybdenum in steels was at one time most frequently carried out gravimetrically as lead molybdate, and this was generally regarded as the standard method, or alternatively, though less often, by precipitation as trisulphide followed by ignition to trioxide. At a later date the colorimetric thiocyanate method by visual comparison involving extraction with butylacetate (or other organic solvent) was utilised for the determination of amounts up to a maximum of 1%. Larger quantities were, and still are, evaluated gravimetrically by the α-benzoinoxime method. The introduction of absorptiometric technique made possible the determination of molybdenum in larger quantities by the thiocyanate reaction, and also dispensed with the extraction of the colour complex by organic solvents. It was found that perchloric-sulphuric acid solutions of certain concentrations were the most suitable for establishing conditions for colour development and stability.[8]

Much work has been published on the photometric analysis of aluminium alloys, and a great deal of attention has been directed to the determination of silicon. The author has developed a method for this purpose (superseding the Regelsburger procedure), utilising an organic reducing agent and dealing with percentages up to 15%.[12]

Passing mention may also be directed to the simplification which can be effected in the evaluation of metallic driers in paints.

An extremely important application of absorptiometric procedure is in the field of micro-analysis, valuable contributions to the literature having been made by Vaughan,[2] Pigott,[13] Belcher[14] and Vaughan and Whalley.[15] Frequent instances of the usefulness of such methods are encountered in modern works' practice. In the early stages of our experience with the absorptiometer the identification of an aluminium alloy, involving the determination of copper, nickel and manganese was readily accomplished on a sample weight of 11 mgms. and analyses of quite a comprehensive character are possible with even smaller weights. The evaluation of small percentages of other elements occurring in high-purity metals is another example of work for which the absorptiometer is particularly well adapted.

7 Stross, W., "Methods of Chemical Analysis of Metals," Amer. Soc. for Testing Materials, 1946.

8 Cox, H., and Pollitt, A. A., "The Photometric Determination of Molybdenum in Metallurgical Products," *J.S.C.I.*, 1944, **63**, 375.

9 Cox, H., and Pollitt, A. A., "Organic Reagents for Metals and for Certain Acid Radicles," Hopkin & Williams, Ltd., 1943, page 141.

10 Phillips, D. F., and Edwards, L. L., "Photometric Analysis—Routine Determination of Copper and Nickel in Aluminium Alloys," *Metal Industry*, 1945, **66**, 409.

11 Cox, H., "The Photometric Determination of Cobalt, Titanium and Iron in Unsintered Metal Carbides," *The Analyst*, 1944, **69**, 235.

12 Cox, H., "The Photometric Determination of Silicon in Aluminium Alloys," *Metallurgia*, 1946, **33**, 121.

13 Pigott, E. C., "Micro-chemical Analysis—Application to Iron and Steel," 1943, 17, 140.

14 Belcher, R., "Micro-chemistry and its Applications," Royal Institute of Chemistry Monograph, 1946.

15 Vaughan, E. J., and Whalley, C., "A New Scheme for the Micro-chemical Analysis of Ferrous Alloys," *Journal of the Iron & Steel Institute*, 1947, **155** (4), 535.

The presence of the so-called " trace " or " residual " metals—e.g., chromium, nickel, molybdenum, copper, etc., in ordinary mild steel is frequently of importance, and it is desirable to be able to determine the quantities present. Most plain carbon steels at the present time contain a certain percentage of these minor constituents, and their determination is very conveniently carried out photometrically, and with greater accuracy and speed than by the methods which were formerly in general use. Procedures for this purpose have been developed and described by Vaughan[2] and Haywood and Wood,[3] and a number of these have been applied by the author.

Determination of Chromium in Steel

For the photometric determination of chromium in steel a procedure was developed which permitted the use of ammonium persulphate as an oxidant, but excluded the addition of silver nitrate. This procedure is based on the fact established by Pigott[16] that chromium can be oxidised quantitatively by ammonium persulphate alone under controlled conditions of acidity. The elimination of the use of silver nitrate as a catalyst prevents the possible introduction of interfering turbidity. A further advantage is gained in that the addition of ammonium persulphate results in complete oxidation and production of clear solutions, subsequent reduction of an aliquot portion providing the blank. The initial solution of the steel in Spekker and nitric acids, with subsequent oxidation of an aliquot portion and retention of the original solution as the blank is not entirely satisfactory, due to the fact that some steels do not furnish clear solutions, even on prolonged digestion in the acid, with the consequence that inaccuracy is introduced.

The method, which follows in detail, has proved satisfactory for the steels normally dealt with in this laboratory, but it is recognised that some steels probably require different treatment at the outset to obtain complete solution. Pigott[17] has investigated this problem in regard to 1% C to 1½% Cr steels for the volumetric determination of chromium, and found that special attention is necessaryto ensure decomposition of the carbides.

Reagents

Spekker Acid	150 mls. Phosphoric Acid (1·75 sp. gr.), and 150 mls. Sulphuric Acid (1·84 sp. gr.) diluted to 1,000 mls.
Nitric Acid	1·42 sp. gr.
Ammonium Persulphate	AnalaR, 25% aqueous solution.
Hydrochloric Acid	20% by volume.
Ferrous Ammonium Sulphate	40 g. dissolved in water, acidified with 10 mls. Sulphuric Acid (1·84 sp. gr.), and diluted to 500 mls.
Manganese Solution	0·75 g. Potassium Permanganate dissolved in water, acidified with 20 mls. Spekker Acid, and reduced with sulphurous acid. Boiled until sulphur dioxide is completely expelled, cooled, and diluted to 1,000 mls.

Procedure :

Cr. max. 6% –1 cm. cell.
max. 1·5%–4 cm. cell.

Weigh 1 g. of the sample into a 250-ml. beaker, dissolve in 20 mls. of Spekker acid, and oxidise by dropwise addition of nitric acid. Digest until the nitrous fumes are expelled, and retain on the edge of the hot plate for approximately 15 mins., then cool. (Solution A).

Cr. max. 20%–1 *cm. cell.*—Dissolve 0·6 g. of the sample as above, transfer the solution to a 100 ml. graduated flask, and dilute to volume with water. Pipette 50 mls. into a 250 ml. beaker (Solution A).

If the manganese content of the steel is known to be low, it is advantageous, at this stage, to add 10 mls. of the manganese solution to Solution A before proceeding. Dilute Solution A to 70 mls. with water, add 20 mls. of ammonium persulphate solution, heat gradually to gentle boiling, and maintain at approximately this temperature on the edge of the hot plate for 10 mins. Add 5 mls. of hydrochloric acid (20%) and again maintain at about boiling-point as before for 5 mins. Cool, transfer to a 100-ml. graduated flask and dilute to volume with water.

Pipette 50 mls. into a 100-ml. graduated flask and dilute to volume. To the remaining 50 mls. add 10 mls. of ferrous ammonium sulphate solution, dilute to volume, and use as the blank. Measure the extinction on the Spekker Absorptiometer, using a size of cell appropriate to the range, and Ilford Spectrum Violet filters No. 601. Obtain the chromium percentage by reference to the graph.

Calibration graph.—Weigh 8·485 g. of potassium dichromate (AnalaR) into a 900 ml. beaker, dissolve in water, add 20 mls. of Spekker acid, and reduce by the addition of sulphurous acid. Boil until the sulphur dioxide is completely expelled. Cool, transfer to a 1,000-ml. graduated flask and dilute to volume with water.

1 ml. ≡ 0·0030 g. Cr.

1 *cm. cell.*—Burette 2–20 mls. of the standard chromium solution in steps of 2 mls. (i.e., 2–20% on 0·3 g. sample) into each of ten 250-ml. beakers. To each add 15 mls. of Spekker acid and 10 mls. of manganese solution, and dilute to 70 mls. with water. Add 20 mls. of ammonium persulphate solution and continue as described under " Procedure." The extinction values are plotted against percentage chromium.

4 *cm. cell.*—Dilute 50 mls. of the standard chromium solution to 200 mls.

1 ml. ≡ 0·00075 g. Cr.

Proceed as in the former calibration, using 4 cm. cells when measuring the extinction values.

Table I gives the results obtained by the above procedure on a number of British Chemical Standard steels, compared to the certificate figures.

TABLE I

B.C.S. Steel	% Cr (certificate figure)	% Cr (Photometric)
165	0·86	0·86, 0·86, 0·85
225	1·04	1·04, 1·05, 1·06
224	1·46	1·46, 1·45
211	13·80	13·63, 13·70, 13·90
209	18·71	18·70, 18·80, 18·90

Composite Method for the Analysis of Welding Flux

Control and development of welding technique and electrode manufacture necessitates analyses on incoming materials and finished welds, and photometric methods have proved of great assistance for this purpose.

It is a well-known practice in the production of alloy steel welds to introduce metallic constituents into electrode fluxes and use a carbon steel core wire. The analysis of such fluxes involving the determination of the loading metals frequently presents difficulties and occupies considerable time. An example of the simplification of this type of work by photometric methods is provided by the following procedure, formulated for the rapid determination of chromium, molybdenum and iron for control purposes. This illustrates how composite methods may be developed for particular requirements and applications, representing a distinct simplification of

16 Pigott, E. C., "The Chemical Analysis of Ferrous Alloys and Foundry Materials," Chapman & Hall, Ltd., 1942.
17 Pigott, E. C., "Iron and Steel Analysis—Some Recent Developments," *Iron and Steel*, 1945, **18**, 641.

analytical procedure, and it will be readily appreciated that it is capable of modification and expansion to include the determination of other metals.

Reagents

Nitric Acid	1·42 sp. gr.
Sulphuric Acid (*a*)	1·84 sp. gr.
Sulphuric Acid (*b*)	50% by volume
Sulphuric Acid (*c*)	10% by volume
Hydrofluoric Acid	40% AnalaR
Perchloric Acid	72% AnalaR
Sodium Bisulphate	AnalaR
Ammonium Thiocyanate	AnalaR, 5% aqueous solution
Potassium Thiocyanate	AnalaR, 20% aqueous solution
Stannous Chloride	AnalaR, 10% in 5% HCl
Ammonium Persulphate, Hydrochloric Acid, Ferrous Ammonium Sulphate, Manganese Solution	as in method for the determination of chromium in steel previously described

Procedure.—Weigh 0·75 g. of the powdered sample into a platinum dish, add 10 mls. of water, 10 mls. of nitric acid, 10 mls. of hydrofluoric acid, and 10 mls. of sulphuric acid (1·84 sp. gr.), and evaporate gradually to fuming. It is inadvisable to fume strongly, otherwise difficulty will be encountered subsequently in effecting solution in water. Cool, add 10 mls. of nitric acid and 10 mls. of hydrofluoric acid, and again evaporate to fuming. Cool, add 40 mls. (approx.) of water, heat to 70° C. and transfer to a 400-ml. beaker. Dilute to 100 mls. (approx.) and digest on the hot plate until all the soluble salts are dissolved.

Filter through a fine filter paper (Filtrate A). Wash with warm water (careful washing of the residue is unnecessary at this stage), transfer the filter-paper and contents to a platinum crucible and ignite. Fuse with 5 g. of sodium bisulphate, cool, transfer to a 250-ml. beaker, and dissolve the melt in 50 mls. of water and 15 mls. of sulphuric acid (1·84 sp. gr.). Filter, wash thoroughly with hot water, discard the residue, and add the filtrate to Filtrate A. Cool, transfer to a 250-ml. graduated flask and dilute to volume with water.

Chromium.—Pipette 50 mls. (0·15 g.) into a 250-ml. beaker, add 10 mls. of manganese solution, and dilute to 70 mls. with water. Proceed as in the method for the determination of chromium in steel described earlier in this article.

Molybdenum.—Weigh 1 g. of spectrographic iron into a 250-ml. beaker and dissolve in 20 mls. of perchloric acid and 10 mls. of water. Into this pipette 25 mls. (0·075 g.) of the flux solution and evaporate to fuming. Cool, dilute with 50 mls. (approx.) of water to dissolve the salts, transfer to a 100-ml. graduated flask and dilute to volume with water. For subsequent procedure, cf., Ref. 8.

Iron.—Pipette two 10 ml. (0·03 g.) aliquots (colour and blank) into 100 ml. graduated flasks and dilute to volume with sulphuric acid (10%). For subsequent procedure, cf., Ref. 11.

It is frequently desirable in photometric work that the addition of reagents be made with accuracy, and 50-ml. burettes graduated at intervals of 5 mls. have proved very useful for this purpose.

Calibration Graphs

In preparing a calibration graph it is the author's usual practice to employ two sources of the metal, and to confirm the calibration by synthetic alloy solutions, or alternatively by standard alloys such as the British Chemical Standard series. It is recommended that extinction readings are repeated until consistency is obtained, and to ensure that the instrument is in a condition of complete stability. This is especially important when conducting a calibration and several readings should be taken at each point.

The graphs which are obtained for one instrument and one set of filters must be retained exclusively for that particular instrument and filters, and must be re-standardised at intervals to compensate for changes which may have occurred in the filters. Such variations may be readily detected by the usual practice of carrying out a determination on an alloy of known composition concurrently with the work in progress.

Filters

The gelatine type of filter is liable to change in spectral absorption characteristics when in use over a period of time, and this lack of stability to heat and light may be noticeable when particular filters have been subjected to constant use. An improvement would probably be obtained by fitting a water-cell to protect the right-hand filter, and an alternative suggestion by Hadley[18] is that provision should be made to accommodate the right-hand filter on the remote side of the calibrated aperture with respect to the lamp.

The protection afforded the left-hand filter by the water-cell and the lack of such protection for the right-hand filter, can, in the author's experience, lead to more pronounced change in the characteristics of the right-hand filter. This may be demonstrated by placing the left-hand filter on the right of the lamp, setting the sensitivity control at full rotation, and opening the drum to give maximum galvanometer deflection. Allowing these adjustments to remain unaltered and replacing the filter by the right-hand one results in a reduced deflection.

This problem would apparently be solved largely by the substitution of glass filters, the use of which appears to find favour in the U.S.A. on account of their greater stability.

Improvements in Operation

The manipulation of the Spekker instrument used by the author has been greatly facilitated by the incorporation of two simple additions. These are of special value in the preparation of calibration graphs and in the development of new procedures. A slow-motion device for operating the drum has been fitted, and consists of a metal frame carrying a shaft provided with a small knurled disc, the frame being bolted to the base-plate of the instrument. The end of the shaft projects beyond the calibrated drum-edge, and carries a rubber sleeve making contact with the knurled edge of the drum. Much greater precision of the drum-setting is thereby obtained by the manual rotation of the small knurled disc on the shaft. A lens has also been placed in the cabinet in such a position as to enable drum-readings to be observed without eye-strain, and this, combined with the slow-motion drive to the drum, permits the readings to be recorded and repeated with great precision. Fig. 1 illustrates the fittings, and is self-explanatory. It is both convenient and efficient to accommodate the instrument and accessories in a suitable cabinet, and the undesirability of location of the instrument on the open bench will doubtless be apparent.

Conclusion

The application of photometric technique still offers wide scope for investigation, and procedures which may appear at the moment to be satisfactory might be

18 Hadley, W. J., "Photometric Determination of Silicon in Aluminium and its Alloys," *The Analyst*, 1945, **70**, 43.

TABLE II

Element	Wt. of Sample in Final Vol. (grams.)	Max. Wt. of Element in Final Vol. (grams.)	Cell	Range (%)
Co	0·0025	0·000375	1 cm.	1·0–15·0
Ti	0·05	0·0075	1 cm.	1·0–15 0
Fe	0·10	0·0010	1 cm.	0·10–1·00
	0·50	0·0010	1 cm.	0·02–0·20
	0·50	0·0005	2 cm.	0·01–0·10
	0·50	0·00025	4 cm.	0·005–0·05
Ni	0·025	0·0010	1 cm.	0·40–4·00
	0·025	0·0005	2 cm.	0·10–2·00
	0·05	0·00025	4 cm.	0·025–0·50
Cr (Dichromate) ..	0·15	0·0300	1 cm.	2·00–20·00
	0·50	0·0300	1 cm.	0·60–6·00
	0·50	0·0075	4 cm.	0·15–1·50
Cr (Diphenyl-carbazide) ..	0·10	0·00025	1 cm.	0·025–0·25
Mo	0·10	0·0010	1 cm.	0·10–1·00
Cu	0·10	0·0010	1 cm.	0·10–1·00
	0·10	0·00025	4 cm.	0·025–0·25
	0·50	0·00025	4 cm.	0·005–0·05
	1·00	0·00025	4 cm.	0·0025–0·025
	2·00	0·00025	4 cm.	0·00125–0·0125
Mn	0·50	0·000625	4 cm.	0·0125–0·125
	1·00	0·000625	4 cm.	0·0063–0·063
	2·00	0·000625	4 cm.	0·0031–0·031
Si	0·02	0·0003	1 cm.	0·10–1·50
	0·002	0·0003	1 cm.	1·00–15·00

examined with accruing advantages. It is emphasised that a high standard of preparatory manipulative work is essential before extinction measurements are taken, and it is highly desirable to be fully cognisant of the most suitable conditions for each determination, and the limiting factors thereof. The necessity of exercising discrimination in the selection of a procedure, including choice of reagents with respect to their various functions, and subsequent critical examination of results is one which justifies reiteration.

It is manifest that in all laboratories constant vigilance must be exercised to eliminate sources of contamination, and it is imperative that scrupulous cleanliness is observed throughout. The ubiquity of two such elements as iron and silicon, particularly in industrial laboratories, emphasises the necessity for maximum precautions to be taken against contamination from glassware, etc., and the laboratory environment.

The quantities of determined elements actually present in the glass cells are, with few exceptions, in the micro category as is shown by consideration of the values in Column 3 of Table II, especially when it is remembered that the capacity of a 1 cm. cell, for example, is approximately 7 mls. Table II gives an indication of some of the ranges most frequently used in this laboratory, and these can be extended, with certain reservations, by varying the size of cell, initial weight of sample and in certain cases the dilution.[3] The range may also be extended in certain cases by using an alternative colour filter. The figures in Columns 2 and 3 refer to weights in the final volume before extinction measurement, which is 100 mls. except in the cases of chromium (diphenylcarbazide)—150 mls. and molybdenum—50 mls.

Acknowledgment

The author is indebted to Mr. A. A. Pollitt, M.Sc., F.R.I.C., for suggestions and advice, and to Mrs. K. Hampson for her careful contributions to the experimental work. He also wishes to thank Sir Arthur P. M. Fleming, C.B.E., D.Eng., Director of Research and Education, and Mr. B. G. Churcher, M.Sc., M.I.E.E. Manager of Research Department, Metropolitan-Vickers Electrical Co., Ltd., for permission to publish this paper.

A Metallurgical Study of German Aircraft Engine and Airframe Parts—Vol. II

In fulfilment of a promise made to the Aero Components Sub-Committee of the Technical Advisory Committee, when the late Dr. T. Swinden was its Chairman, we are publishing the second and concluding part of the Report on German aircraft engine and airframe parts in book form, similar to the copy of the first part published in 1943 and reprinted in 1944. It will be remembered that, in the introduction to the first book it was stated the work included in that report embraced the results of investigations carried out from the beginning of the war until towards the end of 1941, but that the work was continuing. This new report about to be published embraces the results of the further investigations carried out.

The work covered by this report is really a summary of very extensive investigations which have been carried out meticulously and at great cost. Essential information arising from the study of components is presented in concise and readily assimilated form, and the text is supported by numerous illustrations which, in themselves convey valuable information to the initiated. Extensive tabulated data are also presented.

As in the case of Part I of this report, from the proceeds of which £500 has so far been credited to the Royal Air Force Benevolent Fund, we, the publishers, have agreed that the whole of the profits arising from the sale of Part II, will be credited to the same Fund. In order that the maximum sum may become available for such a deserving cause, we are seeking orders for the book in advance of publication. This will enable us to estimate more accurately the number of copies needed before we go to press, and thus limit the number of surplus copies—a waste which would reduce the amount to be credited to the Fund.

The preparation of Part II of this book is well advanced but it is not yet possible to give a publishing date; you may be assured however, that as far as we are concerned there will be no unnecessary delay and orders will be executed as soon as copies are ready. It will comprise about the same number of pages as Part I, and will be worthily bound. Although printing costs have risen since the publication of the earlier book, it has been decided to publish the new book at the same price, viz., 10s. 6d. net.

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MICROANALYSIS
CHEMICAL AND PHYSICAL METHODS
APPARATUS METALLURGICAL APPLICATIONS TECHNIQUE

The Microchemical Balance—Part V

Ancillary Apparatus

By Cecil L. Wilson

Microchemistry Laboratory, Chemistry Department, The Queen's University of Belfast

A provisional list of apparatus which will be employed, sooner or later, by users of the microchemical balance, will already have been visualised by readers of the previous sections of this series. In the present section some attempt is made to detail the varieties of apparatus which may be found to be essential or useful. A considerable proportion of the apparatus can serve purposes other than those closely connected with weighing operations, but it has been considered appropriate to range widely rather than to restrict the discussion to highly specialised apparatus.

In a previous section of this series of articles[1] details were given of the apparatus which should be to hand before dismantling and cleaning the microchemical balance. It will be obvious that a certain amount of this will be in more constant use for the general operation of the balance—for example, the balance case and pans will require daily dusting, and therefore brushes suitable for this purpose should always be available. Likewise, the apparatus for inspection to determine cleanliness—lens and pocket torch—must be at hand. For the present purpose, then, it is assumed that such apparatus already referred to has been acquired. There remains a wide range of ancillary apparatus which has found favour, either as being essential in some form, or as making the operation of weighing more reliable or simpler.

Such apparatus can be broadly divided into two categories: (1) Apparatus concerned with weighing technique, and (2) weighing vessels themselves and their counterpoises.

In the first category, with which the present article is concerned, we must not only consider those materials necessary for ensuring cleanliness of apparatus which is subsequently weighed; it is also legitimate to include the apparatus used in conjunction, both before and after the actual weighing process. Weighing vessels themselves, which will be dealt with separately in a later section, are of divers forms, and often have a number of supplementary functions. Thus we shall have to consider vessels which are solely devised for the actual weighing operation, and vessels in which material is weighed only incidentally in a series of operations, and in which the material is subsequently subjected to further treatment. In addition, the nature of the substance being weighed, whether it is solid or liquid, and, in particular, its hygroscopicity, may have a profound effect on the choice of weighing vessel. Clearly, then, any other than the most specialised laboratory will require that a selection of weighing vessels be available for the varied problems with which the microanalyst may be confronted.

Provision of storage space for such apparatus is a matter which requires early consideration, and, indeed, it should receive serious thought when designing the balance room. No apparatus which can be stored away should be left haphazard about the laboratory or, more particularly, the weighing room. Certain apparatus, it is obvious, must of necessity remain in the balance case. Other of the apparatus will be in general use in the laboratory, apart from its application to weighing technique. But all other apparatus should, as soon after use and cleaning as is convenient, be stored away neatly and systematically. For this purpose, a set of shallow drawers, as near the balance case as practicable, is indispensable.

Perhaps the most satisfactory mode of discussing ancillary apparatus is to follow the operations which comprise the whole weighing procedure, thus referring to each aid in its logical order.

Cleaning Materials

In the first place, any apparatus to be weighed must be cleaned. The preliminary cleaning of apparatus, either by solvents or acids, need not be discussed here, since it involves only general laboratory apparatus. But after preliminary cleaning and drying, glass and porcelain apparatus must be subjected to a standardised wiping procedure,[2] while a certain amount of subsequent handling of the apparatus must be allowed for.

In wiping procedures, moist and dry flannels and

1 Wilson, D. W., "The Microchemical Balance—Part II: Cleaning." *Metallurgia*, 1945, **32**, 85.

2 See—e.g., Belcher, R., and Godbert, A. L., *Semi-micro Quantitative Organic Analysis*, Longmans, 1945; Pregl, F., *Quantitative Organic Microanalysis*, 4th Eng. ed., edit. Grant, J., Churchill, 1945

chamois leathers are usually employed. It will be found most convenient to store these in pairs in Petrie dishes, both to protect them from atmospheric dust, and to ensure that moist wipers remain at a suitable humidity. One pair of moist, and two pairs of dry chamois, approximately 4 in. square, are suitable, housed in three separate Petrie dishes.* These should always be kept in close proximity to the balance case, and should be regularly washed.† Consequently, several spare pairs should be available. If flannel or linen squares are used in place of the moist chamois, these should be free from loose fibres which might stick to the apparatus being wiped. It should hardly be necessary to mention that chamois leathers used for this purpose should be kept distinct from those used for the actual cleaning of the balance.

A probing wire is a useful addition to the cleaning cloths. This is a steel wire, rather less than 1 mm. in diameter, gradually tapered at one end to a blunt point. The tapered end is roughened, as shown in Fig. 1, so that when a small tuft of cotton wool is twisted round it, this adheres tightly, and the wire can then be used in the same fashion as a micro test-tube brush, for cleaning out small-bore tubes (*e.g.*, the ends of absorption tubes) and centrifuge cones or weighing vessels which are long and narrow.

Handling of Apparatus

Handling of apparatus after wiping should always be in such a way as to avoid direct contact with the fingers. Although, as will be mentioned in a moment, a great part of the handling will be by means of forceps, there are occasions when this is not very convenient, and when it may even be clumsy. Therefore, it is useful to have two pairs of chamois finger-stalls, one for each thumb and fore-finger, which may be worn when necessary. These should also be kept scrupulously clean, the washing procedure being similar to that described for the leathers. Finger-stalls cut from surgical rubber gloves may also serve for this purpose, but are rather less comfortable to wear, since in order to ensure that they will stay on they must be rather tight. The consequent effect on the blood circulation in the tips of the fingers may be found unpleasant by the worker.

Handling by finger-stalls is only to be recommended, however, for objects which would be inconvenient to handle by forceps or other similar appliance, and should not be substituted for the latter as a routine procedure.

Platinum-tipped forceps are indispensable for the handling of platinum boats, but will be used for many other purposes, particularly if the inner surfaces of the tips are slightly roughened to prevent too ready slipping on polished glass surfaces. Such forceps may either have solid tips permanently attached, or may be ordinary fine-tipped forceps with little caps, made from platinum foil, fitted over the points. For most purposes the latter are to be preferred, since the inevitable corrosion of the metal of the forceps themselves, after long usage, is not so serious, and can be overcome readily by the substitution of a new pair of forceps. Metal forceps with chamois tips are also useful for handling glass apparatus.[3] Small pieces of chamois leather are stuck to the inside of the tips by a suitable cement,[4] and are then trimmed to shape.

Breuer[5] recommends the attachment of a pair of ordinary bone-tipped forceps to a pair of 24-cm. crucible tongs, so that they can be manipulated from a distance. While such extended forceps can be used for placing weights or counterpoises on the balance pan, a second pair may also be used for the transference of apparatus to the balance, thus avoiding too close approach of the hands to either side of the balance case. This procedure can be recommended, particularly if the balance seems to be unduly temperature-sensitive. It should be emphasised that the pair used for weights and counterpoises should be retained for this purpose only, a separate pair being used for any handling of apparatus.

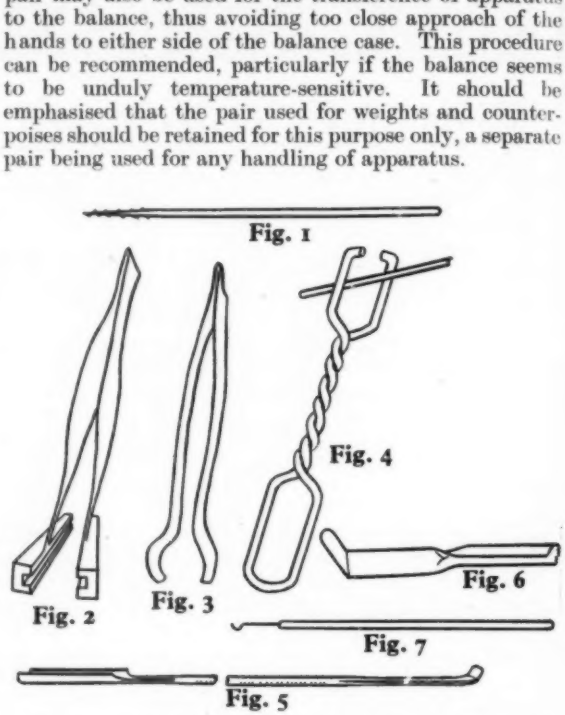


Fig. 1.—Probing wire. Fig. 2.—Forceps for Filter Sticks. Fig. 3.—Forceps for micro-beakers. Fig. 4.—Wire fork. Fig. 5.—Micro-spatula of wire. Fig. 6.—Micro-spatula of foil. Fig. 7.—Platinum hook.

Alber[6] has proposed a special type of forceps for handling filter sticks, since these are liable to slip, even in platinum-tipped forceps with roughened tips. The mode of construction of these, so devised as to grip the stem of the filter stick firmly, will be clear from the sketch of such a pair of forceps in Fig. 2. Forceps with a specially shaped grip for micro-beakers have also been devised, and are illustrated in Fig. 3. Both of these are instances of special cases where, if the special forceps are not available, one may be advised to resort to the use of finger-stalls, since neither filter stick nor beaker is safely handled with ordinary straight-tipped forceps.

Most users of the standard literature in microchemistry will be familiar with the wire forks devised by Pregl[7] for handling absorption tubes. Such forks, being readily made in different sizes, from aluminium wire, will enable other tubular pieces of apparatus to be handled and transported without direct contact with the fingers. A fork is illustrated in Fig. 4.

3 Niederl, J. B., and Niederl, V., *Organic Quantitative Microanalysis*, 2nd ed., Wiley, 1942.

* It is a useful practice to put one of the moist chamois in each of the "dry" Petrie dishes for ten minutes each morning, since it is preferable that the dry leathers should not be too dry.

† Chamois leather may be washed in warm soapy water, and should then be rinsed with dilute ammonia followed by distilled water. After squeezing free of water, the leathers are allowed to dry without artificial heat. The dry leather will be found to be stiff to the touch, but by rubbing it will once again become soft and pliable.

4 E.g., a beeswax-resin cement: see Belcher and Godbert, *loc. cit.*

5 Breuer, F., *Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 354.

6 Alber, H. K., *Mikrochemie*, 1935, **18**, 92.

7 Pregl, *loc. cit.*

Transference of Material

The next operation will probably be the transference of the substance to be weighed to the weighing vessel. Often the precise transference apparatus will depend on the weighing vessel itself, and such specialised cases will be described when discussing weighing vessels. However, general work will require a porcelain or glass tile on which vessels may be placed while solids are being transferred to them. While a white tile is the usual type recommended, the author prefers a black tile, or a tile which is half white, half black. This can be constructed readily by backing a square of plate glass half with white and half with black paper, and protecting this with a coating of cellulose acetate or other suitable plastic varnish. It will be found that on the black half of this tile small particles of material which may have spilled are more readily seen and retrieved. In conjunction with this tile a fine camel-hair or sable pencil is required.

A nickel or platinum spatula of normal design but reduced dimensions is also needed. The author has also used stainless steel, and found this also to be satisfactory for most purposes. A design such as that shown in Fig. 5 is readily beaten out from wire, and can be strongly recommended, the scoop end being particularly valuable. If wire of a suitable thickness is not available, a glass spatula can be made of the same pattern, while something of the same sort can be prepared from thick nickel foil, the stem of the spatula being bent to give it an inverted-V cross section, so as to strengthen it, as illustrated in Fig. 6.

A standard micro-pellet-press, such as that used in micro-ebullioscopic methods for the determination of molecular weight, can prove very useful. Finally, for the transference of liquids, a teat-pipette[8] may be used. This should have a long fine tip, of about 0·25-mm. bore, and in preparing it the walls of the tip should be left as stout as possible.

Heating Processes

From time to time, platinum boats require ignition. A stout platinum hook, inserted in the end of a glass rod, as shown in Fig. 7, is used to suspend the boat in the burner flame, using the small hole in the handle of the boat provided for the purpose. This is preferable to using platinum-tipped forceps. While these latter must be used if the boat is to be held horizontally, continual heating of the tips can rarely avoid some contact of the base metal part of the forceps with the flame from time to time. Ultimately the resultant corrosion causes the tips to break off.

8 Belcher, R., and Wilson, C. L., *Qualitative Inorganic Microanalysis*, Longmans, 1946.

(To be continued)

Microchemistry Group of the Society of Public Analysts and Other Analytical Chemists

AT the Fourth Annual General Meeting of the above Group, the following papers were read.

THE MICROCHEMICAL ASPECTS OF ELECTROLYTIC CONDUCTIVITY

By J. T. Stock

The conductivity of water is markedly affected by traces of dissolved electrolytes, so that the latter may be determined by conductivity measurements. Various cells for the examination of micro-samples have been designed and satisfactory results are obtainable if the ancillary apparatus and conditions are suitable. Since the end-point in conductometric titration is located graphically, useful results are obtainable when the titration curve is distorted by effects such as hydrolysis, solubility of precipitate, etc. The technique is thus applicable at the low concentrations sometimes encountered in microchemical work. Various special techniques such as differential titration, selective precipitation, application of high-frequency oscillators, etc., have been developed, and may prove valuable on the microchemical scale.

A MICRO BLOWPIPE FOR THE MANIPULATION OF CAPILLARIES

By J. T. Stock and M. A. Fill

The blowpipe, which gives a small, hot flame, may be constructed in various forms for use on the bench or in the hand. Principles of construction are, however, similar throughout. In its simplest form, the device consists of a steel washer, on the opposite faces of which are soldered discs of copper foil. The burner orifice and air jet are formed simultaneously by puncturing both discs with a fine sewing needle. Tubes attached to the rear disc permit the entry of air and gas. Compressed air is supplied by a mercury-piston pump operated from the water supply.

A DIAPHRAGM PUMP FOR AIR AND OTHER GASES

By J. T. Stock and M. A. Fill

The pump is actuated by an electric bell movement, the pump mechanism being held by the screw which normally retains the gong. As the hammer vibrates, it repeatedly depresses a thin rubber diaphragm stretched across the mouth of a thistle funnel. The stem of the latter is bent and is attached to a chamber containing a pair of disc valves, so that air is drawn in at the lower and forced out at the upper end of the chamber. Though simple, the pump is useful for circulating gases, providing an air stream for stirring in micro-titrations, for pumping hydrogen sulphide from a suitably designed generator and similar purposes.

A MELTING POINT INDICATING DEVICE

By J. T. Stock and M. A. Fill

Owing to the necessity for constant observation, the accurate determination of melting points is tedious. By employing a fine glass rod pressing lightly upon the sample at the bottom of the melting-point tube, sintering incipient to melting is indicated by a slight sinking of the rod. The latter carries at its upper end a light platinum bridge which makes contact with a pair of mercury cups when the rod sinks. The circuit thus completed sounds a buzzer or other audible warning, so that the actual melting may be observed visually. Tiny beads of

coloured glass fused to the lower ends of the melting-point tube and the rod considerably facilitate the visual observation.

A TRANSMITTING MANOMETER FOR MICRO OXYGEN UPTAKE EXPERIMENTS

By J. T. Stock and M. A. Fill

On the micro scale, the capacity of a manometer compared with that of the remainder of the system is not negligible. It is, therefore, desirable to thermostat the entire apparatus, such as by operating in a constant-temperature oven. To follow pressure-changes, an electrical method is used. The mercury manometer is of usual appearance, but has fine platinum filaments stretched centrally in either limb. Together with a pair of external resistances, the filaments form a Wheatstone network, the state of balance of which is continuously indicated by a galvanometer outside the oven. Alterations in pressure alter the effective lengths, and hence resistances, of the filaments in the circuit, so that the galvanometer reading changes accordingly.

An exhibition of new microchemical apparatus designed by the above authors was also held.

Polarographic Discussion Panel

A MEETING of the Polarographic Discussion Panel of the Physical Methods Group of the Society of Public Analysts and Other Analytical Chemists was held in the Concert Hall, L.C.C. Norwood Technical College, London, S.E. 27, on Friday afternoon, December 12th, 1947, when a discussion on "The Validity of the Ilkovic Equation in Polarographic Analysis" was opened by Mr. F. L. Steghart.

Having surveyed the existing work which appears to support the Ilkovic equation, Mr. Steghart described experiments using a super-fast recording instrument, the current passing through the solution being amplified without distortion and fed into a pen recorder. Changes in current throughout the life of the individual mercury drops could thus be traced. Unlike the conventional damped circuit normally used in polarography, the time-lag in these experiments was never more than $\frac{1}{10}$ of a second.

If the faraday-requirement per mole, the diffusion coefficient and the concentration of the depolariser, together with the rate of mercury flow, be assumed to be constant during the life of any one drop, then the Ilkovic equation may be simplified to :

$$i_t = kt^{1/6} \qquad (1)$$

where i_t is the current flowing at time t and k is a constant obtainable experimentally by examining known solutions. The current should thus vary as the $\frac{1}{6}$ power of the time, but the curves obtained did not fit this relationship. Replacing $t^{1/6}$ by t^z, where z is to be calculated (1) may be rewritten :

$$z = \frac{\log i_t - \log k}{\log t} \qquad (2)$$

The values of z obtained varied between the approximate limits of 2/3 at the initial stages in the life of the drop to 1/3 at the end.

The average current, I, is defined by :

$$I = \frac{1}{t_{max}} \int_0^{t_{max}} i_t dt \qquad (3)$$

Conventional damped instruments tend to take less account of the short period when the current is low than of the long period when the current is high, thereby giving an average reading which is approximately 14% too high. By combination of equations (1) and (3) and subsequent integration, the ratio I/i_{max} comes out to be 6/7. If, however, in the light of the experimental evidence, a value of $t^{1/3}$ is used in place of $t^{1/6}$, the ratio is $\frac{3}{4}$—i.e., about 14% lower. By chance, this difference is opposite and about equal to the instrument error of the conventional instrument, so that with the latter compensation occurs and this may explain why the Ilkovic equation appears to be obeyed.

During the life of the drop, the current increases directly with the drop surface area, S, so that the relationship :

$$i \propto S \propto kt^{2/3} \qquad (4)$$

should hold. By cine-photographing the growth of a drop and computing the surface area from enlargements of each frame, it was found that the area-time relationship was not quite true at the beginning of the drop-life. The error however should tend to increase the rate of change of current in the early stages, whereas the previous results show that the rate of change compared with that calculated from the Ilkovic equation is too small. This fact enlarges the discrepancy between actual current and that calculated from the Ilkovic equation.

Mr. Steghart concluded by pointing out that the above anomalies do not detract from the utility of the polarograph in chemical analysis, since calibration methods are usually employed. In fundamental work, the current which flows immediately before the fall of the drop should be measured, since it is subject to the least instrument error. So far, all the work described (with the exception of the photography of the drop) has been carried out by Mrs. B. Lamb in the laboratories of Tinsley (Industrial Instruments), Ltd. Both he and Mrs. Lamb propose to continue their experiments, but would be pleased to co-operate with others interested.

The discussion then ranged over various aspects of the subject, such as the work of Lingane *et al.* on the effect of the magnitude of the mass of mercury flowing per second, possible equations alternative to that of Ilkovic, etc. Among those taking part were Mrs. Lamb, Drs. Kalousek and Page, and Messrs. Smith and Stock.

Institution of Metallurgists

The next examinations for the Licentiateship and Associateship of the Institution of Metallurgists will commence on September 6th, 1948. Candidates may submit their applications for *permission to enter* at any time before April 1st, 1948. Each application must be made on a form to be obtained from the Registrar, Institution of Metallurgists, 4, Grosvenor Gardens, London, S.W.1, and must be accompanied by a registration fee of one guinea. The remainder of the examination fee will be payable not later than August 1st, 1948, by candidates whose applications to enter for the examination are accepted. The registration fee will not be returnable in any circumstances. Applications received after April 1st, 1948, will not be considered.

Ceramic Materials for High-Temperature Mechanical Parts

By Joseph R. Bressman

ONE of the important material advances of recent years has been the development of high strength ceramic materials for high-temperature service. The coming of the jet engine, gas turbine, rocket, and the atomic energy plant, with their demands for new and better high-temperature materials, stimulated this development. Ceramics possess a number of inherent advantages over metals. Their melting points, in general, are much higher. Their bulk density is considerably lower. And they are very hard and chemically inert, therefore, more resistant to erosion and corrosion.

The main purpose of American research in this field has not been to substitute ceramics for metals in the range 1,300°–1,500° F., but to develop ceramics that will permit operations at temperatures above 1,500° F. The investigations have proceeded along two chief lines: the development of solid ceramic shapes for high temperature parts; and the development of ceramic coatings to protect metals from corrosion at high temperatures.

The first requirement of a ceramic is that it must have sufficient strength to support tremendous centrifugal loads. In this respect ceramics have an inherent advantage over metals. The centrifugal force in a rotating blade is proportional to the density of the material; the higher the density the greater the tensile strength must be. The density of high-temperature ceramics varies from 2·5–5, as compared to 7 or 8 for high-temperature alloys. Closely related to this strength requirement are the stress-rupture and creep strength properties. For aircraft use the stress-rupture strength is used as a design criterion. For example, the material may be designed for 500–1,000-hr. life for transport service, 150 hr. for military pursuit planes, or even 1–4 hr. for a guided missile engine.

The creep strength is used as a design criterion for such purposes as marine, locomotive and stationary power plants. The load to cause 0·1% elongation in 10,000 or 100,000 hr. is often used.

Gas turbines, superchargers and jet engines rotate so rapidly that the materials pass through millions of stress cycles in a very short time; consequently, the fatigue strength is of importance for any application except, perhaps, guided-missile application.

In addition to the extraordinary demands on stress-rupture, creep and fatigue strength, the material must be resistant to mechanical and thermal shock to an unusual degree. Other requirements of ceramics for gas turbines are similar to those for metals.

The most important high temperature ceramics at present are compositions that contain combinations of refractory oxides of the following elements: beryllium (BeO), aluminium (Al_2O_3), zirconium (ZrO_2), thorium (ThO_2), magnesium (MgO), and calcium (CaO). Typical bodies developed contain large percentages of beryllium oxide and zirconium oxide, with smaller quantities of oxides of magnesium, calcium, silicon, titanium or boron. Older refractory bodies contained large proportions of mullite ($3Al_2O_3\ 2SiO_2$).

Short-time tensile and stress-rupture tests were conducted on specimens prepared from a sillimanite ore by the hydropress method. The specimens were fired at 3,100° F., and consisted largely of mullite and free silica in the form of cristobalite. The results of the short-time tensile tests show that the tensile strength at 1,800° F. was greater than the room temperature strength and exceeded 18,000 lb./sq. in. It was also noted that specimens heated for ½ hr. at 1,800° F., and cooled slowly, exhibited higher tensile strengths up to 1,400° F. than specimens not heat-treated.

The results of stress-rupture tests are given in Table I. The stress-rupture strength decreased rapidly from 1,400°–1,800° F., but the density is only one-third that of high temperature metals.

Much more encouraging stress-rupture and creep results were achieved on new compositions. The bodies tested were simple compositions of refractory oxides whose main constituents were BeO or ZrO_2. The specimens were also hydropressed in rubber moulds. The best results in creep tests were obtained on a composition which consisted approximately of 84 BeO, 7 each of Al_2O_3 and ZrO_2, and 2% CaO. The bulk density 1,800° F. by increasing the stress from 9,000–14,000 lb./sq. in. at intervals of approximately one week. The total test time on the single specimen, including preliminary tests at various lower temperatures, was 2,589 hr. The test was discontinued after 117 hr. at 14,000 lb./sq. in. because the thermocouples became unreliable. The creep of this specimen was the lowest of all those tested at 1,800° F. At 2,100° F., however, the deformation was excessive. The results on this body may be summarised by noting that satisfactory resistance to creep and rupture were achieved at a stress of 14,000 lb./sq. in. at 1,500° F., 13,000 lb./sq. in. at 1,800° F., and 4,000 lb./ sq. in. at 2,050° F. This composition is better than ordinary metals at 1,800° F. on a straight strength comparison, and is far superior when compared on a strength-density ratio basis.

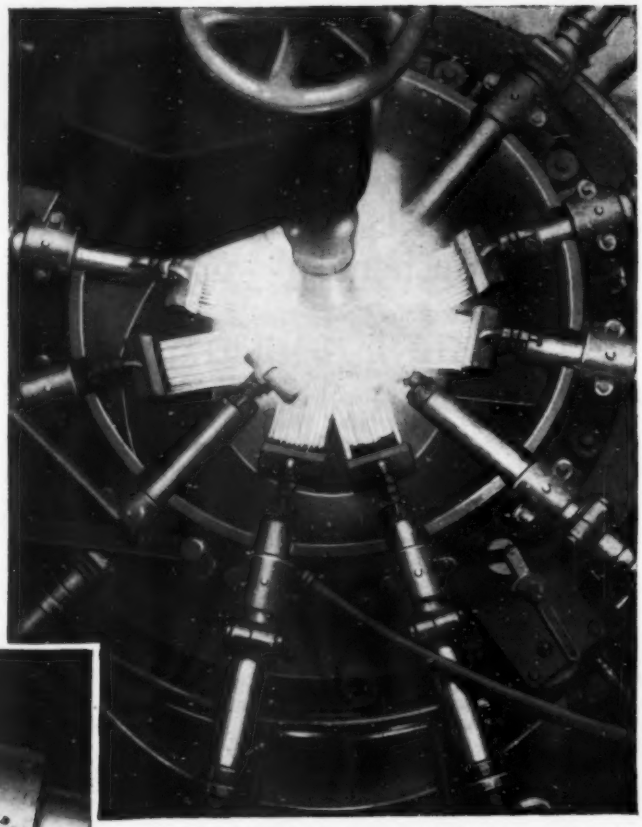
TABLE I.—STRESS-TO-RUPTURE TESTS ON SILLIMANITE REFRACTORY

Temperature (F.)	Stress (Per sq. in.)	Time to Rupture (Hr.)
1800	9400	0·42
	8500	1·17
	6700	19·1
1600	9600	99·2
	8600	722
1400	6750	522*
	7700	242*
	8650	212*
	9600	251

* Specimen did not fail under these stress conditions; at the end of this time the stress was increased to the next higher increment.

From *Materials and Methods*, 1947, **27**, 65-70

MAKE SHORTER WORK OF IT WITH OXY-ACETYLENE FLAME HARDENING



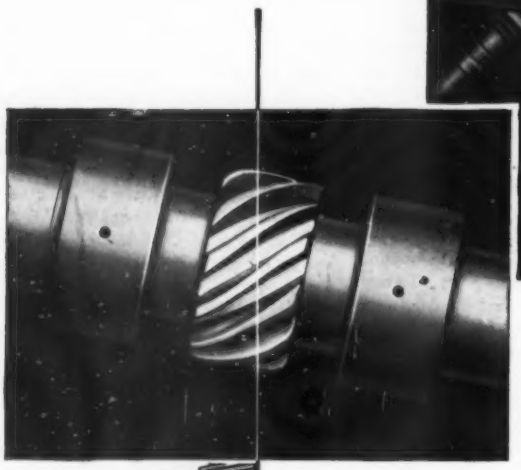


Illustration by courtesy of Head Wrightson & Co., Ltd.

Hardening once required a whole series of operations—but the Shorter Flame Hardening Process reduces the number. By heating just that part of an article which needs to be hard for maximum wear resistance, and immediately quenching the heated area with a jet of water, the B.O.C. Shorter Flame Hardening machines enable gear teeth, for example, to be treated in one pass of the oxy-acetylene flame. The top illustration shows a roll hardening machine in operation. The roll is rotated vertically, and the flame and quenching heads of the machine travel along its length. The inset shows the areas hardened on the teeth of a typical gear—the roots of the teeth and the core of the gear remain tough and ductile—the flanks of the teeth are hardened in just the right place. For full technical data, write for Booklet TIB/13.

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The high-temperature refractories can be formed into various shapes by many methods. Complicated shapes can be made by "slip casting." In this method a plaster mould of the object is made. A thin paste or slip of the ceramic and water is then poured into the hollow mould. The plaster absorbs water from the slip depositing a layer of the ceramic on the mould. When the proper thickness has built up, the excess is drained off. The object can then be dried, removed from the mould and fired.

Objects of high strengths can be made by dry pressing in which practically dry ceramic powders are compacted under pressure in a mould. The piece is then removed and fired in a furnace. An interesting method of accomplishing this is a process known as "hydropressing," in which the powder is placed in a rubber mould of the proper size and shape and then subjected to hydrostatic pressure. When the "green" ceramic is removed from the mould it is firm enough to be machined or ground.

Ceramic shapes can also be made by extrusion through dies. Another useful method of fabrication is casting from the molten state. Large glass tank blocks are made in this way by melting mullite at temperatures over 3,300° F. and casting the molten liquid into special moulds.

Where fine tolerances are necessary the dry pressing methods, such as hydropressing, are very useful. The "green" or unfired object can be moulded to the proper size, taking into account shrinkage in the furnace. Objects have been made this way in to tolerances of $\pm 0{\cdot}002$ in. Where closer tolerances are necessary, surfaces must be ground after firing.

Refractory Coatings for Metals

Ceramic coatings for high temperature use require several special characteristics that are peculiar to their application. A coating must, of course, adhere well to the base metal over the full range of temperatures to which it will be submitted. This means that the rate of thermal expansion must be quite similar to that of the base metal. The coating should have high resistance to thermal shock and temperature gradients. A continuous sealed surface must be maintained over the metal in order to protect it from oxidation. Finally, the material should have a low solubility for metals or their oxides and should not "reboil." Reboil is the property that results in the appearance of gas bubbles in the coating and usually occurs on reheating after the first firing operation.

The coatings are prepared in similar manner to that for porcelain enamels. The base metal must be properly cleaned and free from grease. It must be pickled or, if possible, sand-blasted and given a nickel flash. The coatings are usually sprayed on, but can be applied to interior surfaces by dipping. The coatings are then dried and fired. Single coatings are of the order of $0{\cdot}002$–$0{\cdot}004$ in. thick. The firing temperature with ordinary steels is 1,500°–1,600° F.

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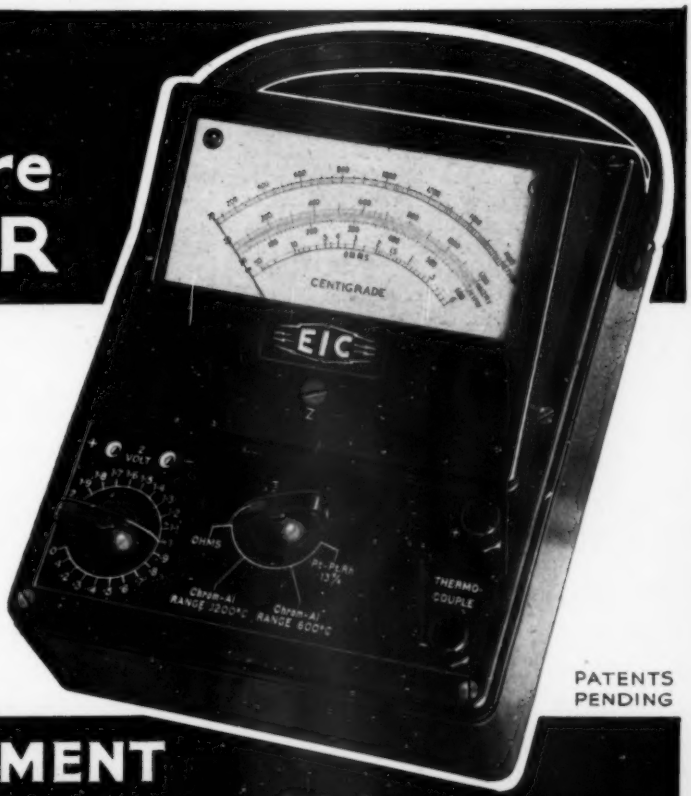

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The effect of a coating on the room temperature tensile strength of low alloy steels that were heated for 500 hr. in air at 1,200° F. is as follows: ingot iron, coated specimen strength, 225% greater than uncoated specimen; SAE 1010, 55% greater; SAE 1020, 25% greater; SAE 4130, 16% greater. The coatings on the last two specimens were slightly blistered, which reduced their effectiveness. Similar tests were conducted on high-temperature alloys at 1350° F. in an atmosphere of hydrogen sulphide, which was found to be particularly corrosive. The results of these tests are indicated in Table II.

Service tests have been conducted on aircraft and tank exhaust manifolds and collector rings, turbosupercharger blades, jet engine and buzz bomb flame tubes. The coatings proved so successful for exhausting systems that they were used in regular production for many Army and Navy applications. The ceramic coatings of the jet engine flame tubes doubled the life of the tubes in service tests. Buzz bomb flame tubes ran cooler and were not subject to heat warping.

The new refractory coatings, although showing great promise and potentialities, have some limitations. The temperatures at which the coatings can be used have a potential limit in that the firing temperatures must increase with increased refractoriness, but obviously cannot exceed the melting point of the base metal.

Industrial applications might be furnace parts, high-temperature heat exchangers, chemical processing apparatus, smoke stacks, hot gas ducts, steam turbine blades, nozzles, piping and other steam power plant equipment.

TABLE II.—EFFECT OF CERAMIC COATING ON TENSILE STRENGTH

Alloy	Coating	Time at 1350°F. (Hr.)	Yield Strength at Room Temp.
S-816	None	471	Specimen broke before testing
S-816	Alumina type	471	69,000 average
S-590	None	189	Specimen broke before testing
S-590	Alumina type	189	70,000 average
S-590	None	101	33,000
S-590	Cobalt oxide type	101	76,000

Aluminium Collapsible Tubes

AN account is presented of the origin, output figures, advantages and the production of aluminium collapsible tubes. American domestic shipments early in 1947 had increased 237% over a comparable first-quarter period in 1937. Over half a million gross of aluminium tubes had been produced in the first eight months of 1947, with an anticipated three-quarter million or more gross to be shipped before the year is out. This, in effect, represents a 20% increase in domestic aluminium tube shipments over 1946.

At present, according to the Aluminium Co. of America, aluminium collapsible tubes are being used for packaging more than 70 diversified products. The Aluminium Co. of America operates a packaging laboratory at Edgewater, N.J., devoted exclusively to the testing of prospective customers' materials. Aluminium tubes filled with the product it is intended to package undergo a rigorous 90-day trial period. The tubes and their contents are stored at approximately 100° F., and are examined periodically for distortion, internal gas pressure or leakage at the crimped ends. The interior tube surfaces are examined for any evidence of pitting or staining. The contents are carefully checked against control samples for appearance and colour. Only after these tests have been completed, and tubes and product passed, is the laboratory permitted to approve the combination.

Internally coated aluminium tubes are still in the development stage. Three basic types of interior coating for aluminium tubes have been developed. One is a compounded natural resin, another is a synthetic resin, and the third is a compounded-tempered amorphous wax. All are inert to destructive acids and alkalis and are inherently non-toxic.

Production of aluminium collapsible tubes starts with the blanking of circular slugs from aluminium plate varying from $\frac{1}{8}$–$\frac{1}{4}$ in. in thickness. There follow the impact extrusion, trimming and threading operations, after which a conveyor carries the tubes through washing equipment which sprays them under pressure with a detergent, removing lubricant and surface dirt. The tubes are left spotlessly clean and ready for annealing which is carried out at 1,100° F. The surface of the annealed tubes is in excellent condition to receive an enamel base coating. The enamel is applied by menas of enamelling rolls, the tubes being carried past them on revolving mandrels. After enamelling, the tubes are placed on pin conveyors and sent to drying ovens, where the enamel is baked to a lustrous finish. Offset printing on the enamelled tubes is done in a similar way.

Filling, closing and sealing equipment for aluminium tubes is relatively inexpensive. Hand-, semi-, and fully-automatic types are available, depending upon a packager's own production requirements. Final crimping of tubes may vary from a double- or quadruple-fold chipless closure for non-seeping products, such as pastes and adhesives, to a double-fold, heat-sealed closure for products where temperature increases may produce expansion.

Packaging Parade, November, 1947; *Alcoa Digest*, **17**, No. 25, pp. 1–3, December 8th, 1947. Reproduced from *Light Metals Review*, 1948, **7**, 20–1.

Aluminium for the Canning of Food

By R. CHEVILLOTE.

THE corrosion resistance of aluminium and the colourless and non-toxic properties of its salts add to its suitability for use in food canning. Small quantities of aluminium salts occur naturally in some fruits and vegetables and it has been shown that their presence in food is harmless. Impetus was given to the application of aluminium in France by the shortage of tin during the war. The fitting of airtight tops by means of soldering is not practicable with aluminium cans and two types of covering are used: the cap type fitting over the can and the inserted lid type. The construction and suitability of both types are outlined.

Despite its high corrosive resistance, aluminium is sometimes attacked by substances having a high acid or sodium chloride content. Such attack is accompanied by the evolution of gas which gives rise to bulging. The importance of determining the acid content in relation to both corrosion and the temperature of sterilisation is stressed. The method of heating produce before packing it into cans is considered to be particularly suitable where aluminium is used. Shell fish, sardines and chestnut puree are amongst the products which have been successfully canned. To prevent any attack which might occur when aluminium is exposed for long periods to sweetened evaporated milk, the Swiss have made use of anodic oxidation.

Revue de l'Aluminium, 1947, 24, 100–108. Abstract from *Light Metals Review*, VI, No. 18, 381.